Diamond & Related Materials xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

Diamond & Related Materials

journal homepage: www.elsevier.com/locate/diamond

Characterization of the recovery of mechanical properties of ion-implanted diamond after thermal annealing

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ARTICLE INFO

Article history:
Received 4 August 2015
Received in revised form 17 November 2015
Accepted 17 November 2015
Available online xxxx

Keywords:
Diamond
Ion-induced damage
Ion beam lithography
Mechanical properties
Atomic Force Microscopy

ABSTRACT

Due to their outstanding mechanical properties, diamond and diamond-like materials find significant technological applications ranging from well-established industrial fields (cutting tools, coatings, etc.) to more advanced mechanical devices as micro- and nano-electromechanical systems. The use of energetic ions is a powerful and versatile tool to fabricate three-dimensional micro-mechanical structures. In this context, it is of paramount importance to have an accurate knowledge of the effects of ion-induced structural damage on the mechanical properties of this material, primarily to predict potential undesired side-effects of the ion implantation process, and possibly to tailor the desired mechanical properties of the fabricated devices. We present an Atomic Force Microscopy (AFM) characterization of free-standing cantilevers in single-crystal diamond obtained by a FIB-assisted lift-off technique, which allows the determination of the Young's modulus of the diamond crystal after the MeV ion irradiation process concurrent to the fabrication of the microstructures, and subsequent thermal annealing. The AFM measurements were performed with the beam-bending technique and show that the thermal annealing process allows for an effective recovery of the mechanical properties of the pristine crystal.

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1. Introduction

MeV ion implantation has been widely exploited in recent years for the micro-fabrication of single-crystal diamond, through the implementation of the so-called "lift-off technique" [1–4]. This technique can be effectively adopted to fabricate micro-mechanical structures in single-crystal diamond, with applications ranging from high-frequency MEMS devices [5–11] to opto-mechanical resonators [12], thus taking advantage of the extreme mechanical properties of diamond [13]. Recently, the latter topic attracted significant interest due to the outstanding properties of nitrogen-vacancy centers in diamond [13], whose spin-dependent optical transition can effectively couple with local mechanical stresses [14–16]. To this end, various different techniques have been employed to fabricate opto-mechanical resonators in diamond [17–20].

In the case of the lift-off technique, the fabrication process is based on the local conversion of diamond to a sacrificial graphitic layer through MeV-ion-induced damage and subsequent thermal annealing [4]. The fabrication technique is very versatile, because the local induced damage density can be controlled by varying implantation parameters (namely ion energy, species and fluence). Nonetheless, a residual damage density (and related mechanical stress) is induced in the non-sacrificial regions as a side-effect of the fabrication technique [21]. Similarly, with other fabrication techniques [17–20], a residual damage can be induced in the fabricated opto-mechanical microstructures, particularly if ion implantation is adopted to induce the formation of nitrogen-vacancy centers [22].

For these reasons, it is necessary to accurately estimate deformation and stress levels to reliably design and fabricate MEMS structures. Moreover, the variation of elastic properties of damaged diamond as a function of induced damage density and post-processing (annealing) parameters remains to be clarified. In particular the Young's modulus of ion-implanted diamond can potentially vary between that of pristine diamond (>1 TPa, in the presence of no damage) and that of amorphous carbon (~10 GPa, for full amorphization), i.e. over two orders of magnitude. Clearly, this large variation in elastic properties is likely to strongly affect modeling results in the fabrication of mechanical and opto-mechanical resonators. Attempts have been made to experimentally derive the variation of elastic properties of diamond as a function of induced damage, but only indirect estimations with limited accuracy have been obtained [23]. This lack of experimental data is partly due

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http://dx.doi.org/10.1016/j.diamond.2015.11.008
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to its high Young's modulus, which makes it difficult to perform indentation experiments. Here, we perform a study of the elastic properties of ion-implanted and subsequently annealed single-crystal diamond by means of Atomic Force Microscopy (AFM) measurements on free-standing cantilever structures microfabricated with a FIB-assisted lift-off technique [3,4].

2. Micro-fabrication

An artificial diamond sample grown by High Pressure High Temperature (HPHT) by ElementSix (UK) was employed in this work (product code: 145-500-0040). The sample is $2.6 \times 2.6 \times 0.5$ mm^3 in size and is classified as type Ib, on the basis of a nominal concentration of substitutional nitrogen <200 ppm. As indicated by the producer, the sample typically consists of ~80% single <100> sector, thus the effects of growth-sector boundaries on its mechanical properties are negligible. It is known that HPHT diamond samples contain catalyst impurities such as iron, nickel or cobalt, typically at concentration levels of the order of ~10 ppm [24–26]. At such concentrations, the effect of metallic impurities on the mechanical properties is negligible, and the Young's modulus of these "mechanical grade" crystals is comparable to that of CVD-grown samples (i.e. ~1.1 TPa [27]). The sample is cut along the [100] crystal direction and is optically polished on one of the two opposite large faces. The sample was implanted at room temperature across one of the polished surfaces with 800 keV He$^+$ ions at the AN2000 accelerator of the INFN National Laboratories of Legnaro with a focused ion beam, in order to deliver a fluence of 1×10^{17} cm^{-2}. The microbeam spot was ~10 μm in diameter, and was raster-scanned to implant a rectangular area of ~500 × 200 μm^2. The high density of damage induced by ion implantation promotes the conversion of the diamond lattice into an amorphous phase within a layer which is located at ~1.4 μm below the sample surface, as shown in Fig. 1.

The sample was then annealed in high vacuum (~10^{-6} mbar) at 1000 °C for 1 h, to convert the highly-damaged regions located at the end of the ion range to a graphitic phase while removing the structural sub-threshold damage introduced in the layer overlying the damaged region. Following the fabrication scheme described in [1], FIB milling with 30 keV Ga$^+$ ions was subsequently performed on the implanted area, to expose the sub-superficial graphitic layer to the subsequent etching step, while defining the geometries of three different cantilever structures characterized by different widths (see Fig. 2). A thin Au film was deposited on the sample surface to avoid charge effects during FIB micro-machining. The sample was then exposed to contact less electrochemical etching [30]: the sample was placed for several hours in deionized water with the region of interest comprised between two adjacent (i.e. few millimeters) Pt electrodes kept at a DC voltage difference of ~100 V. The process resulted in the selective removal of the sacrificial graphitic layer and in the creation of free-standing cantilever structures with a lateral geometry defined by the previous FIB micromachining, i.e. a length of 117 μm for cantilevers #1 and #2 and of 111 μm for cantilever #3. The widths of cantilevers #1, #2 and #3 were respectively 13 μm, 9 μm and 22 μm. The thickness of all cantilevers corresponded to the penetration depth of the employed 800 keV ions, i.e. 1.3 μm. As shown in Fig. 2, all cantilevers are slightly bent upwards due to residual stresses induced by the fabrication process within the "cap layer" comprised between the sub-superficial graphitic layer and the sample surface. In previous works this effect has been observed in structures fabricated with the lift-off method [31] and is due to the inhomogeneous swelling through the depth of the beam caused by the non-uniform ion damage profile, as shown in Fig. 1. Thermal annealing is known to reduce this effect, but it does not remove entirely the influence of residual defects. This material swelling at the underside of the beam can also cause a slight local increase in the beam cross section, however the effect is negligible with respect to the FIB-machining related effects, which are discussed below.

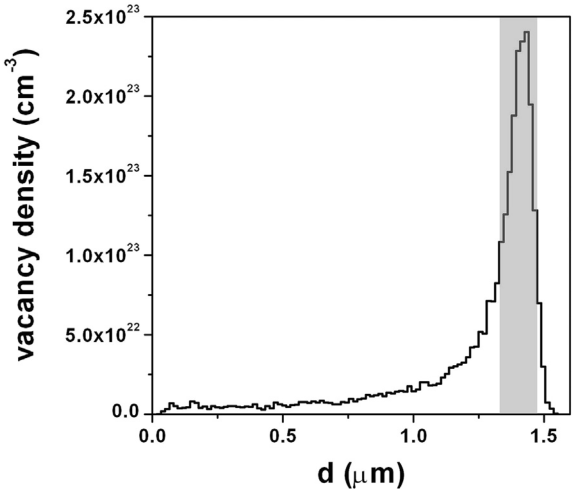

Fig. 1. Depth profile of the vacancy density induced by 800 keV He$^+$ ion implantation at fluence 1×10^{17} cm^{-2}, as evaluated with SRIM2013.00 Monte Carlo code [28] and assuming a linear dependence of the induced vacancy density from the implantation fluence [29].

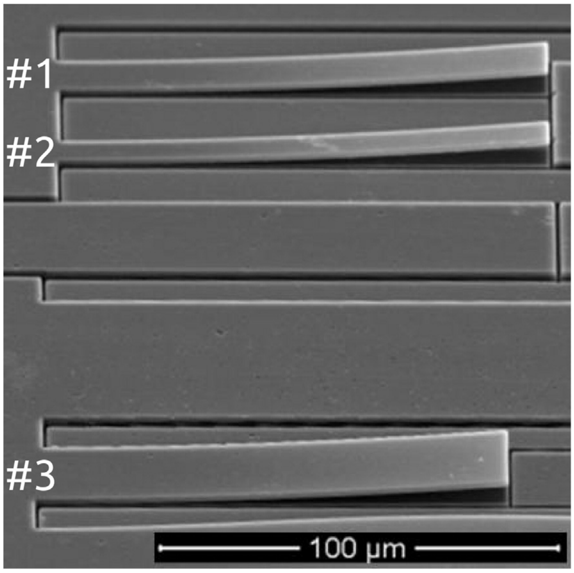

Fig. 2. SEM micrograph of the three free-standing cantilever structures fabricated in single-crystal diamond by means of the FIB-assisted lift-off technique.

3. AFM characterization

In order to determine the Young's modulus of the diamond, a beam-bending method is employed [32–34]. The method consists in loading the microstructures using an AFM cantilever. As shown in Fig. 3, the deflection d of the probing AFM cantilever for the displacement z of the piezomotor is measured by means of a laser diode and a position-sensitive photodiode (Veeco Dimension 3100).

The effective stiffness k_{eff} of the system based on the coupling of the two cantilever structures is measured by recording approach curves. The effective stiffness k_{eq} is equal to:

$$k_{eff} = \frac{1}{\frac{1}{k_{AFM}} + \frac{1}{k_{diam}}} \tag{1}$$

Please cite this article as: M. Mohr, et al., Characterization of the recovery of mechanical properties of ion-implanted diamond after thermal annealing, Diamond Relat. Mater. (2015), http://dx.doi.org/10.1016/j.diamond.2015.11.008

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