Determination and tuning of Young's modulus modification in ion-implanted diamond

A. Battiato[1,2,3*], E. P. Ambrosio[4], E. Bernardi[1,2,3], F. Bosia[1,2,3], M. Lorusso[5],
P. Olivero[1,2,3], F. Picollo[3,1,2], D. Ugues[5]

[1] Physics Department, University of Torino, Torino, Italy,
[2] NIS Inter-departmental centre, University of Torino, Torino, Italy,
[3] INFN Sezione di Torino, Torino, Italy,
[4]Department of Applied Science and Technologies, Politecnico di Torino, Italy,
[5]Center for Space Human Robotics @Polito, Istituto Italiano di Tecnologia, Torino, Italy.
*battiato@to.infn.it

INTRODUCTION

MeV ion implantation has been widely exploited in recent years for the micro-fabrication and functionalization of single crystal diamond. Various works have shown that this technique can be effectively adopted to engineer the electrical, optical and structural properties of diamond [1-2]. The induced damage density can be controlled by varying implantation parameters, such as ion species and fluence, resulting in the formation of point defects (essentially vacancies and interstitials), in the amorphization and eventually in the permanent graphitization of the pristine crystal upon thermal annealing when a critical damage threshold is reached [3].

It is known that mechanical deformation (surface swelling) effects are associated to the structural variations occurring in the crystal due to ion implantation. One issue that remains to be adequately addressed, however, is the variation of elastic properties of damaged diamond as a function of the implantation fluence, in particular that of its Young's modulus, which is expected to vary between that of pristine diamond (>1 TPa, in the presence of no damage) to that of amorphous carbon (~10 GPa, for full amorphization), i.e. by two orders of magnitude. Attempts have been made to experimentally derive the variation of elastic properties, e.g. in [4], but only indirect estimations with limited accuracy have been obtained. This lack of experimental data is partly due to diamond's extremely high values of Young's modulus (the stiffest known material, together with carbon nanotubes or graphene), which makes it difficult to perform indentation experiments.

Here, we perform a systematic study of the elastic properties of ion implanted diamond by means of nano-indentation measurements.

MATERIALS AND METHODS

The sample under exam is a synthetic IIa single crystal diamond produced by chemical vapour deposition. The diamond is $3\times3\times0.3$ mm^3 in size, cut along the (100) crystalline direction with four optically polished faces: two opposite large faces and two opposite small faces.

The sample was implanted at room temperature with a 2 MeV H^+ ion microbeam at the ion microscopy line of the AN2000 accelerator facility of the INFN Legnaro National Laboratories (INFN-LNL). A rectangular area was implanted with the fluence of $1\cdot10^{17}$ cm^{-2} on the small polished face in proximity of the edge between this face and the large one. This implantation geometry allows to measure the mechanical properties (i.e. Hardness and Young's modulus) of the damaged region at increasing ion penetration depth by means of a nano-indentation profilometer on the unimplanted polished face (see **Fig. 1a**).

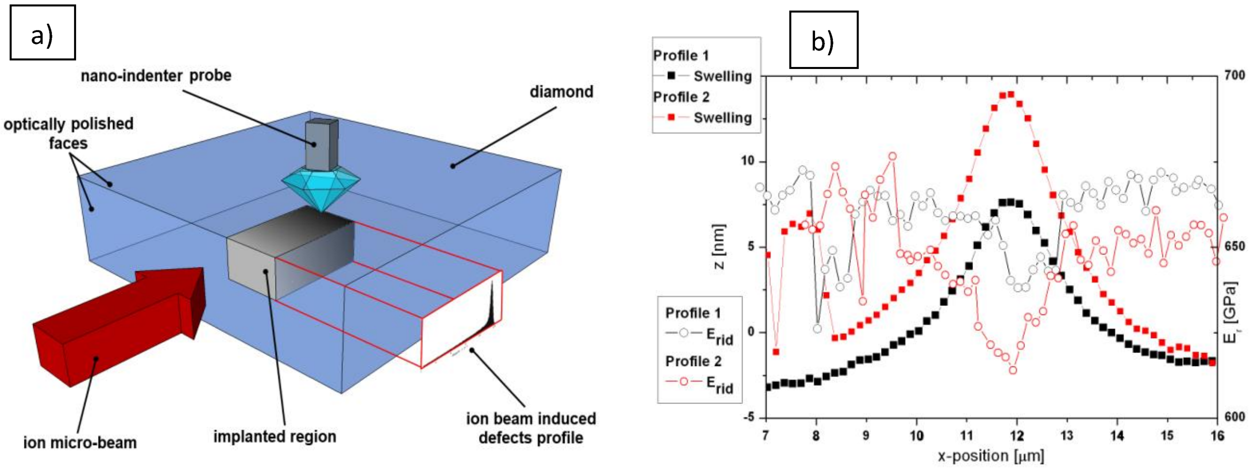

Fig. 1: **a)** schematic of the experimental set up; **b)** topography and reduced modulus (E_{rid}) profiles

Nanoindentation is an indentation test in which the length scale of the penetration is measured in nanometers. The distinguishing feature of most nanoindentation tests is the indirect measurement of the area between the indenter and the specimen (contact area). The contact area is determined by measuring the depth of penetration of the indenter into the specimen surface. During the nanoindentation test, a force versus displacement curve is produced applying a force by the transducer and observing the resulting displacement. The analysis of these curve provide information regarding the mechanical properties of the sample, in particular on Reduced Modulus (E_r). In this work nanoindentation tests were carried out with an Hysitron instrument (TI 950 TriboIndenter), using a Berkovich tip. A Berkovich tip is a 3-sided pyramid with an included angle of 142.3°, where the angle from the normal to a face is 65.35°.

Profiles were realized at a distance of 5 µm, each profile was realized with 80 indents at a distance of 0.15 µm. A load of 5000 µN was applied with a rate of 500 µN s^{-1}, the maximum load for each indentation is applied for 2 s.

RESULTS AND DISCUSSION

In **Fig. 1b**, red and black circles represent the values of Reduced Modulus collected along to different scan lines. In both cases a minimum in the Reduced Modulus for the same x-position can be noted. These minima in Reduced Modulus are localized in the implanted area, as can be seen comparing E_r profiles with topography profiles (red and black squares); in fact, the minima in Reduced Modulus and the maxima in topography (swelling) occur at the same x-position. E_r values far from the implanted area agree with the one of pristine diamond.

CONCLUSIONS

These preliminary results indicate that nanoindentation is an insightful technique to directly probe the elastic properties of ion implanted diamond. Future activities will be focused on the fine tuning of the Young's modulus; ab-initio and FEM simulations are also foreseen in order to better understand this softening mechanism.

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