Optical Characterization of Proton Irradiated Diamond

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ABSTRACT

Manufacturing of miniaturized photonic devices based on diamond technology is possible by implanting the pristine material with highly energetic particles. Here we report on the spectral characterization of the optical constants of proton-irradiated diamond. Absorption of the irradiated zones was estimated in the UV-vis-NIR from direct transmittance measurement using a dedicated setup with enhanced spatial resolution. The OPD data providing an estimation of the thickness of the damaged area and its depth profile, have allowed then evaluation of the extinction coefficient from the transmission measurements. Simultaneous variation of dispersive optical constants makes the modeling significantly more complicated compared to the above cited monochromatic study.

Keywords: Optical constants; Diamond crystal; Ion implantation

1. INTRODUCTION

Manufacturing of miniaturized photonic devices based on diamond technology is possible by implanting the pristine material with highly energetic particles. Theoretically the optical constants of irradiated material may vary from the values typical for pristine diamond to those of graphite, however only precise knowledge of the optical constants allows calculation of performance of the novel photonic micro- and nano-devices like e.g. diamond-based waveguiding structures[1].

The geometry of the damaged zones represents the principal obstacle for their characterization. Their lateral dimensions, in fact, render hardly possible implementation of commercial spectrophotometers, while on the other hand an adequate modeling is necessary to describe variation of optical constants within the specimen depth. The refractive index of proton-implanted zones in CVD-grown diamond have been estimated recently at 632.8 nm from interferometric measurements of optical path difference (OPD) and a model has been developed for description of the in-depth damaged material as a stack of stratified media composing an inhomogeneity profile[2].

Here we further develop the argument and report on the spectral characterization of the optical constants of proton-damaged diamond. Absorption of the irradiated zones was estimated in the UV-Vis-NIR from direct transmittance measurement using a dedicated setup with enhanced spatial resolution. The OPD data providing an estimation of the thickness of the damaged area and its depth profile, have allowed then evaluation of the extinction coefficient from the transmission measurements. Simultaneous variation of dispersive optical constants makes the modeling significantly more complicated compared to the above cited monochromatic study.

Section 2 is dedicated to the experimental details of sample preparation and characterization. Section 3 describes the obtained results and discusses the particularity of the optical constants modeling.

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2. SAMPLE PREPARATION AND CHARACTERIZATION

The samples under study are type IIa single-crystal diamonds grown by Chemical Vapour Deposition (CVD) technique by ElementSix[3]. The single {100} grown sectors of the size 3.0x3.0x0.5 mm^3 were cut along <100> axes and their faces were optically polished to guarantee Ra surface roughness of 2 nm. As specified by the produced, the concentration of nitrogen and boron impurities are below 0.1 ppm and 0.05 ppm, respectively.

On the polished surface of the samples a raster-scanning proton beam was then applied for implantation over the square areas of about 125x125 µm^2 (external scanning microbeam facility of the LABEC[4]). Figure 1 shows a photo of one of the samples under study. The beam was extracted into a helium atmosphere to minimize lateral and energy straggling and had focus diameter at the sample surface of ~20 µm and ~10 µm for proton energies of 2 and 3 MeV, respectively. Real time monitoring of the beam charge was performed by measuring the X-ray yield from the beam exit window, ensuring a 3% accuracy of both beam charge and fluence determination. For different implantations (different zones over the same sample) the fluence values varied from ~10^{13} cm^{-2} to ~10^{17} cm^{-2}, while the ion current varied between 0.2 nA and 1.5 nA.

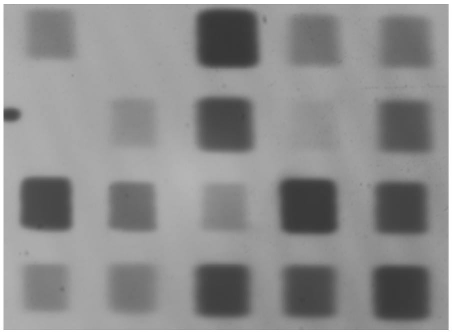

Figure 1. Transmission optical image of several 125×125 µm^2 implanted areas.

A laser interferometric microscope (Maxim 3D, Zygo Corporation) operating at wavelength of 632.8nm was employed[2] for mapping of optical path difference (OPD) with micrometer lateral resolution and sub-nanometer accuracy in the optical thickness direction. The field of view was 349×317 µm^2 with an optical resolution of 1.68 µm, and the interferometric resolution in the thickness direction was 0.63 nm, so that the OPD signal was relevant to the whole sample thickness crossed by the test beam, and it was not affected by optical absorption phenomenon. The change in OPD is to be attributed to the change in optical thickness with respect to the unimplanted surrounding area, hence to a change in the material refractive index. In the cited study, the maximum variation of the refractive index amounts to ~0.1, corresponding to ~4% of the absolute value at λ=632.8 nm (n=2.41).

The transmittance spectra were acquired by a home-made setup for measurement with enhanced spatial resolution[5]. The schematic representation of the setup is given in Fig.2.

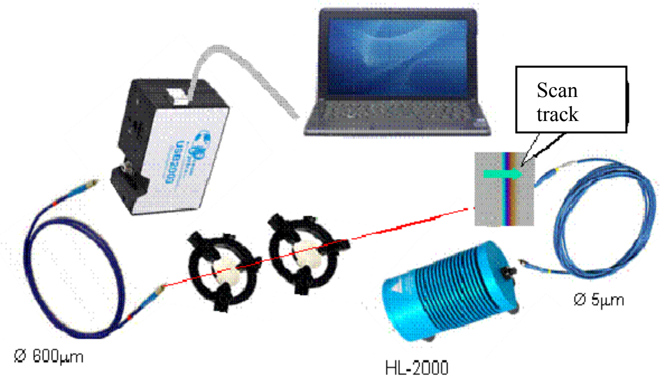

Figure 2. Scheme of the transmittance measurement set-up.

The size of the irradiated zones guarantees that for characterization of the modified material it is sufficient to reduce the diameter of the illuminating beam spot down to 100 μm, if consider slight curvature of the swell at the plateau borders. Nevertheless, a smaller beam spot would keep from broadening of possible spectral peculiarities, if a local change of transmission is very steep both spectrally and spatially. On the other hand, the mechanical stage of the setup allows sampling with a minimal step of 25 μm, and this value has been used to individuate a precise location of the center of the irradiated zone that was supposed to correspond to minimum of local transmission. The above considerations made us to choose the spot size of about 50 μm for characterization of these samples. The implemented spectrometer covers the spectral range of 400-1200 nm with 0.8 nm sampling. Taking into consideration the chosen spatial resolution of the setup, the spectral resolution may be ensured to be of at least 1 nm. However, the acquired transmission spectra are free from narrow peaks or other types of steep slopes, thus even lower spectral resolution would be sufficient for this application.

3. TRANSMITTANCE MEASUREMENT ELABORATION

Theoretical modeling of inhomogeneous dispersive ion-implanted material as a stack of several mono-material strata each thick of several micrometers is based on SRIM [7] simulation of the damage profiles for implanted zones, Fig.3. This approach has allowed a reasonable simplification of description of a complex three-dimensional geometry of the irradiated zone.

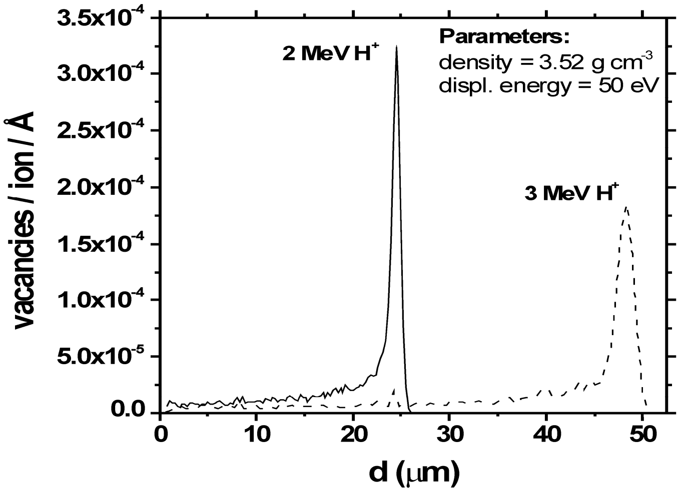

Figure 3. SRIM simulation of diamond damage profiles towards the specimen depth, for implantations of protons with energies of 2MeV and 3 MeV.

A schematic representation of the model is shown in Fig.4. The validity of such a model for the implanted depth, where the absence of interlayer interference is guaranteed by micrometric thickness of each stratum, has been shown in Ref.[2] for the monochromatic case of 632.8 nm. In particular, of the variation Δn of the refractive index from the value of that of pristine diamond at λ=632.8 nm, was shown to depend on the vacancy density ν as $\Delta n = (4.29 \pm 0.05) \times 10^{-23} \times \nu[\mathrm{cm}^{-3}]$. This dependence has allowed to obtain the initial approximation for the refractive indices and extinction coefficients of the irradiated material as following. The refractive index of each of the five strata was designed as that of beforehand characterized pristine diamond corrected to an additional value calculated using the above formula. The resulted values at 632.8 nm are reported in Fig.4.

Note that the traditional approach of the spectral curve fitting by a modeled multilayer with varying thicknesses and varying optical constants is hardly usable for this application. In fact, the number of variables and their range of uncertainty (especially, for the thickness of the strata) are too big and make uncertain the convergence of an algorithm to a unique reasonable solution.

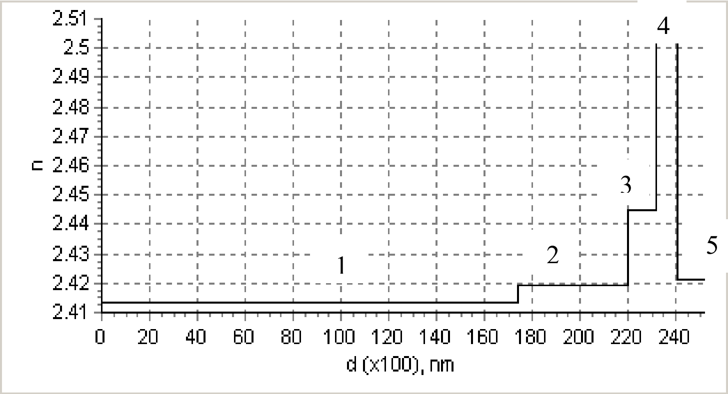

Figure 4. Profile of refractive index at 632.8nm – initial approximation for the fit - of the area irradiated at 2 MeV proton energy.

The abundant literature on the diamond and diamond-like materials provides the optical constants dispersion laws for the CVD-grown diamond and the amorphous carbon films (see e.g. [6]). Cauchy type dispersion is the natural way to describe the strata refractive indices, when their variation from that of the pristine material does not exceed 4%^{2}:

$$n_i(\lambda) = n_{0i} + \frac{A}{\lambda^2}.$$

Here i indicates the stratum index, and n_{0i} and A are the two fitting parameters. However, considering the strata thicknesses, it is worth to keep the first approximation (left panel of Fig. 5) for the dispersion curves invariable in order to prevent the problem instability, while searching for the shape of the k dispersion curves.

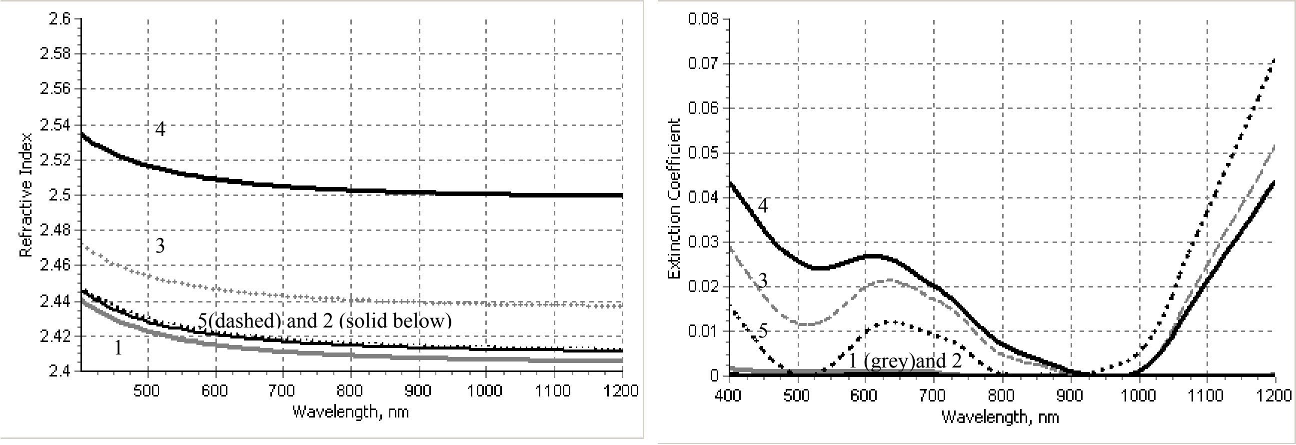

Figure 5. Refractive indices (left) and extinction coefficients (right) of the five strata composing the area irradiated at 2 MeV proton energy with fluence 1x10^{16} cm^{-2}.

Right panel of Fig.5 shows a solution for the extinction coefficients of the five strata within one of the irradiated areas of the sample. Initial approximation for this fit of k was constant in wavelength values representing the absorption level in each stratum proportional to the vacation density. The solution shown in Fig. 5 maintains this physical meaning and provides a very good fit, Fig. 6.

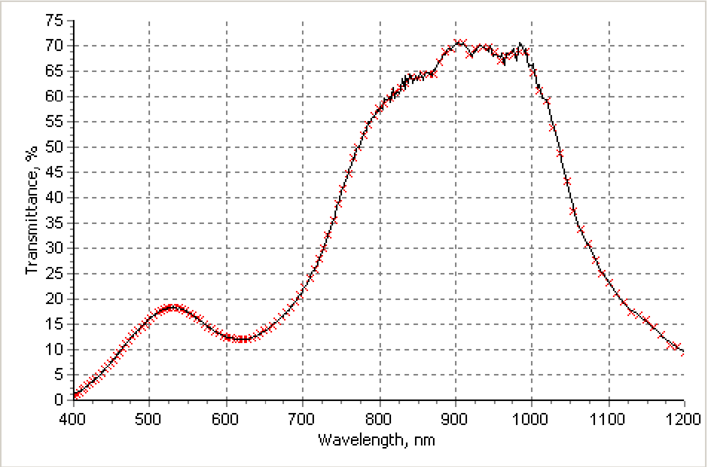

Figure 6. Simulated (solid line) and measured (crosses) transmittance of the area irradiated at 2 MeV proton energy with fluence $1x10^{16}$ cm^{-2}.

However it is possible to obtain a similarly good fit for other values of extinction coefficients of the five strata. In particular, the solution search procedure tends to converge automatically to much lower values of k while attributing to thicker strata higher absorption, like in the case of the stratum 1, Fig. 7. This can not correspond to a physical solution, when the far more damaged stratum 4 results just slightly more absorptive compared to the others and, moreover, having qualitatively different type of k dispersion.

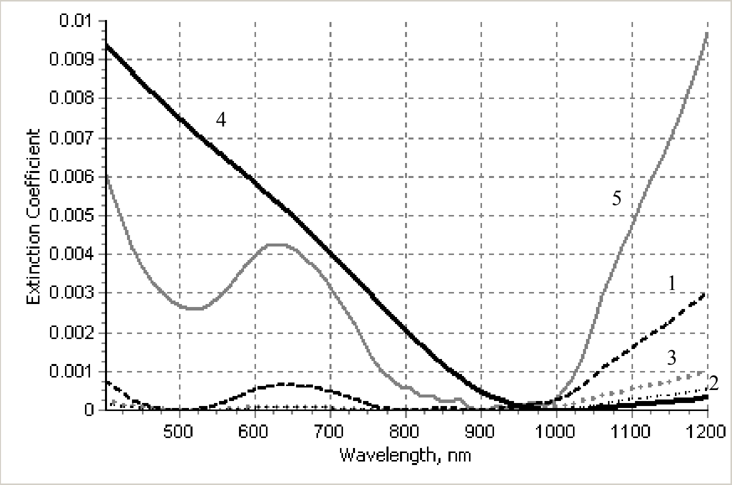

Figure 7. Other possible mathematical solution for the extinction coefficient of the five strata composing the area irradiated at 2 MeV proton energy with fluence $1x10^{16}$ cm^{-2}.

4. CONCLUSIONS

Careful modeling of the initial approximation of the optical constants of proton-irradiated diamond is necessary to obtain physically meaningful results. The characterization example of a multistratum having complex geometry is given in this work. The extinction coefficient of the materials composing the irradiated area follows similar wavelength dependence showing a double-oscillator absorption in the Visible (centered at 630 nm and 720 nm approximately) and a strong increase towards both UV (down from 500 nm) and IR (for wavelength longer than 1000 nm). In the range 500-1100 nm, however, the irradiated material results rather transparent even for relatively high values of radiation fluence. A complete study on the dependence of the induced absorption level on the irradiation conditions will be published in a separate work.

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