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Microsphere lithography for scalable polycrystalline diamond-based near-infrared photonic crystals fabrication

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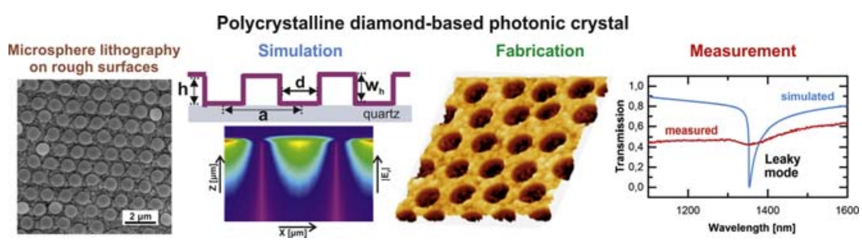
HIGHLIGHTS

- Microsphere lithography employed on rough surfaces
- Fabrication of hexagonal air-hole-type diamond structures on large areas
- Diamond 2D photonic crystals for near-infrared telecommunication wavelength region

GRAPHICAL ABSTRACT



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ABSTRACT

We demonstrate the fabrication of a polycrystalline diamond-based two-dimensional photonic crystal (PhC) slab using microsphere lithography (MSL). First, the essential issues of MSL on rough diamond surfaces are demonstrated. The peak-to-valley value of the rough surfaces is shown as the most important parameter in choosing the appropriate sphere size for the MSL technique. Second, we fabricate a diamond-based PhC composed of a hexagonal lattice of air holes and tuned to have a leaky mode in the near-infrared wavelength region at 1.31 μm which is an important telecommunication window. A monolayer of plasma-treated polystyrene microspheres with controllable size and periodicity, combined with a metal mask, are utilised as templates in the fabrication process. The prepared diamond films and photonic crystal are characterised using scanning electron microscopy, atomic force microscopy, Raman spectroscopy, reflection, and transmission measurements. In order to investigate the optical properties of the PhC, the experimental measurements are compared with simulation outputs. The successful employment of MSL on rough diamond films with their unique properties opens the road to the large scale fabrication of highly durable and chemically inert scalable structures.

1. Introduction

Diamond has gained reputation as an exceptionally versatile material due to its attractive physical and chemical properties, such as robustness, low dielectric constant and loss, high thermal conductivity, and biocompatibility [1–3]. Furthermore, diamond has a wide bandgap (5.5 eV) with the strongest UV emission line positioned at 5.27 eV (235 nm) at room temperature, high refractive index (2.41), wide transparency window from deep UV to the far IR, and can host different colour centres (e.g. nitrogen, silicon, erbium) [4–7]. These fascinating properties make diamond an ideal material for multispectral optical applications, including photonics [8]. Although single-crystal diamond is a better candidate for optical devices than polycrystalline diamond, the production and machining of single-crystals is quite cumbersome [9]. In contrast, polycrystalline diamond film with desired properties (thickness, morphology, grain size) can be easily and inexpensively deposited on a wide range of substrates and over large areas [10]. It also retains many of the unique characteristics of single-crystal diamond. Certainly, polycrystalline diamond has

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its drawbacks. Its slightly poorer optical properties are a consequence of light scattering due to surface roughness, absorption losses, and nonuniform optical properties due to the presence of non-diamond carbon phases located at the grain boundaries [11,12]. Despite these limitations, polycrystalline diamond has also been successfully used as an eligible material for the realisation of various optical applications [13–16].

Photonic crystals (PhC) are periodic structures artificially designed in one-, two- or three-dimensions for monitoring, controlling or modification of light inside the structure [17]. One of the most important applications of the 1D and 2D PhC structures in photonics is a very efficient coupling of incident light from outside into a planar waveguide. The same structure can also be used reciprocally, i.e. as an efficient light outcoupler. Both these effects appear thanks to the existence of the so-called leaky modes. Leaky modes are modes of the PhC that are extracted to the surroundings by the Bragg diffraction on the periodicity. Wavelength and angle of the extracted light are given by the geometry and dimensions of the PhC. PhCs with two-dimensional periodicity can also be employed as polarization splitters [18]. However, the fabrication of such periodically arranged uniform nanostructures on large areas (~cm^2) is a challenging task. This is particularly true in the case of diamond. Due to its extreme properties (i.e. chemical inertness, high-temperature stability and extreme hardness), it is difficult to structure diamond [19].

Nowadays, various lithographic techniques are used for fabrication of micro- or nanoscale diamond structures in a controlled fashion [20]. Although they have almost reached perfection, most of them (e.g. electron-beam lithography (EBL) or focused ion beam lithography) suffer from high fabrication cost and relatively long fabrication time, especially for processing large areas or structures with large dimensions [21]. Thus, it is highly desirable to develop an effective, easy and scalable method to sculpt diamond or other material-based periodic structures (e.g. photonic crystals, cavities, etc.). Nano- or microsphere lithography (MSL) has a great potential as an effective alternative to aforementioned lithographic techniques [22]. It is regarded as a material independent fabrication approach to manufacture a wide variety of 1D–3D tailor-made periodic structures, i.e. create nanostructures with alterable and definable characteristics (different feature size, shape, lattice constant, etc.) [23,24]. Thereby, it also offers the possibility to realise diamond photonic structures with dimensions tuned for the extraction/coupling of specific wavelengths, such as 738 nm (emission band of SiV centres), 1.31 and 1.55 μm (telecommunication windows).

The principle of MSL is based on the self-assembly of monodisperse microspheres (e.g. polystyrene (PS), SiO_2, metallic, etc.) into a hexagonal close-packed (hcp) monolayer or face-centered cubic multilayer [25,26]. These arrays of spheres are used as masks or templates for various subsequent deposition, etching or imprinting processes [27–29]. Nowadays, it is successfully used for surface patterning over large areas. However, it is primarily considered for flat or polished surfaces. Here we extend the applicability of MSL on rough polycrystalline diamond film.

The aim of this paper is to combine microsphere lithography and diamond growth technology to fabricate a two-dimensional polycrystalline diamond-based PhC. First, we describe the technological setup and discuss the fabrication challenges of the MSL especially focusing on the diamond film surface roughness and PS microspheres diameter. Next, we present a simple step-by-step procedure for creating a large area diamond PhC with hexagonal lattice on a quartz substrate. The desired dimensions of the structure are given by computer simulations. Finally, we compare the simulated and measured transmission spectra of fabricated diamond-based PhC tuned for extraction/coupling of 1.31 μm wavelength (the second optical communication window).

2. Experimental part

2.1. Microsphere lithography on rough polycrystalline diamond films

First, several experiments were performed to demonstrate the applicability of MSL on polycrystalline diamond films with various morphological properties. In this case, the high diamond film surface roughness is the main obstacle to achieve a high quality hexagonal close-packed monolayer mask.

Three series (labelled as A, B and C) of diamond thin films were deposited with varying growth times (1, 6 and 12 h) using microwave (MW) plasma enhanced chemical vapour deposition. The other growth parameters were as follows: MW power 3 kW, pressure 6 kPa, 1% CH_4 in H_2, temperature 750 °C. Next, monolayers of PS spheres (with diameters of 0.5 and 1 μm) were created on diamond films using spin-coating technique with optimised process variables. Beside these, dispersion with a very low concentration of PS spheres was drop-casted onto other diamond films to compare the size of the spheres and diamond grains.

2.2. Diamond photonic crystal design and fabrication

2.2.1. PhC design and simulation

A two-dimensional diamond-based air-hole-type PhC on a quartz substrate with hexagonal lattice symmetry was designed. The desired dimensions of the PhC, i.e. thickness of diamond film (h), lattice constant (a), diameter (d), and depth of holes (w_h), were estimated by simulations using rigorous-coupled wave-analysis (RCWA) technique implemented in software RSoft DiffractMOD.

2.2.2. PhC fabrication

The proposed PhC was fabricated through seven technological steps. Their schematic illustration is shown in Fig. 1. The desired dimensions of the structure were chosen based on the results of the simulations.

1) ***Diamond nucleation and deposition***: Ultrasonically cleaned mirror-polished quartz (JGS1) of size 1 × 1 cm^2 was used as substrate. It was nucleated using ultrasonic seeding with diamond nanoparticles (NanoAmando Aqueous Colloid: Dispersed Buckydiamond with a median diamond grain size of 4.8 ± 0.6 nm). Polycrystalline diamond film was grown in a focused microwave plasma enhanced chemical vapour deposition reactor (Aixtron P6). Process parameters used for the deposition process were as follows: 3 kW, 6 kPa, 1% CH_4 in H_2, 750 °C, 1 h.
2) ***PS monolayer formation (mask preparation I)***: After the growth process, the diamond film was treated in oxygen plasma to achieve hydrophilic surface. Next, a monolayer of polystyrene microspheres was formed using three-step spin-coating process. The polystyrene particles were purchased from microparticles GmbH as a 10 wt% in dispersion with standard deviation ~0.03 μm. The initial diameter of the PS microspheres was a = 1080 nm, which defined the lattice constant of the photonic structure.
3) ***PS etching (mask modification)***: Reactive ion etching (RIE) was applied to reduce the PS microspheres' initial diameter to the desired one. The modified (i.e. non-close packed) monolayer acts as a physical mask for further processing. The plasma treatment was performed in pulsed MW linear antenna plasma system (AK400, Roth & Rau) in oxygen plasma (300 W, 100 Pa, 100 sccm O_2, 20 min). More details about plasma etching of PS spheres can be found in Ref. [30].
4) ***Au evaporation (mask preparation II)***: Thin Au film (60 nm) was evaporated onto the modified sphere array.
5) ***PS removal***: After gold deposition, the PS mask was removed by gentle ultrasonication in toluene.
6) ***Diamond etching***: The air holes in diamond films were created using RF plasma etching (Plasmalab System 100, Oxford Plasma Technology). The holes depth was determined by process parameters: 100 W, 7 Pa, O_2:CF_4 = 45:2, 10 min.
7) ***Au removal***: The final diamond PhC structure was obtained by a chemical etching of the Au film in Potassium Iodide solution.