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Development of nanocrystalline diamond windows for application in synchrotron beamlines

R.A. Campos [a,*], V.J. Trava-Airoldi [a], O.R. Bagnato [b], J.R. Moro [c], E.J. Corat [a]

[a] INPE, Instituto Nacional de Pesquisas Espaciais, LAS, CP 515, 12227-010 São José dos Campos, SP, Brazil
[b] LNLS, Laboratório Nacional de Luz Síncrotron, CP 13083-970 Campinas, SP, Brazil
[c] IFSP, Instituto Federal de Educação, Ciência e Tecnologia de São Paulo, CP 770, 12929-600 Bragança Paulista, SP, Brazil

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ABSTRACT

A multistep growth and masking method allowed developing windows with controlled geometry inside a silicon frame. In this paper, we present a new method to produce nanocrystalline diamond windows with thickness of about 200 nm to 40 μm, with different areas and shapes (circular, rectangular and rounded rectangle). The nanocrystalline diamond (NCD) films deposited on a silicon substrate (100) p-type, had a nucleation density of about 10^{11} part/cm^2. Electrostatic self-assembly of nanodiamond seeds (4 nm powder) improved nucleation. Silicon anisotropic etching reveals the window geometry. The high nucleation density enabled smooth surfaces on both sides without the need for polishing the window. Pressure tests were performed in windows of varying thickness. The windows with thickness larger than 10 μm supported a pressure gradient of 1 atm.

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1. Introduction

CVD diamond has been applied as window material in X-ray synchrotron beamlines [1–3]. The main advantage of nanocrystalline diamond (NCD) films is the low roughness. The microcrystalline diamond (MCD) presents considerable roughness for application in synchrotron beamlines and need polishing. The window roughness influences directly the spatial coherence of the beam [4]. Further, the defects propagate more easily in the grain boundaries of MCD films. This characteristic directly influences the mechanical properties of the films [5].

In general, these windows need materials with low atomic number to transmit more electromagnetic radiation. Beryllium (Be) is the main choice material, but with limited transmission in the visible range. On the other hand, the diamond has transparency from the visible to the X-ray [6]. One characteristic of CVD diamond is to keep most of the properties of natural diamond. Besides a wide transparency range it also has the great advantage of a high thermal conductivity [7]. The thermal conductivity of diamond (1000–2000 W/m K – depending on the quality) [8] is about ten orders of magnitude higher than the beryllium (~201 W/m K). This set of properties associated with the mechanical characteristics of diamond films makes it as an excellent candidate for applications in optical windows. Recently published works have explored the multispectral properties of CVD diamond, to develop windows and filters for X-rays [9], and focal lenses for lasers [10].

Successful nanocrystalline diamond film deposition with homogeneous and nanometric thickness depends on high rate of film nucleation. Over the years various studies comparing the nucleation mechanisms have been presented [11,12]. Among the most efficient nucleation mechanisms we highlight the processes aided by bias enhanced nucleation (BEN), in which the nucleation densities are close to 10^9 part/cm^2 [13,14]. However, it is proven that BEN incorporates diamond nuclei in a matrix of amorphous carbon [15]. The presence of amorphous carbon affects the film quality. A new method recently published in the literature called ESND (Electrostatic self-assembly seeding of nanocrystalline diamond) explored the electrostatic interaction between the substrate and the diamond nanoparticles in the seeding process [16]. This is an adaptation of the ESA (electrostatic self-assembly) method [17]. This method is based on a multilayer construction by electrostatic attraction between opposite charges on each layer. The nucleation density with this method is better than BEN with the advantage of an amorphous carbon free layer. From ESND the nucleation densities were around 10^{11} part/cm^2.

In this paper we developed freestanding nanocrystalline diamonds windows grown by Hot Filament Chemical Vapor Deposition

* Corresponding author. MCT – INPE – Circuito Impresso, Av dos Astronautas, 1758 – Jd. Granja, CEP: 12227-010, São José dos, SP, Brazil. Tel.: +55 12 32086571; fax: +55 12 32086717.
E-mail addresses: campos@las.inpe.br (R.A. Campos), corat@las.inpe.br (E.J. Corat).

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(HFCVD), with thickness as small as 200 nm and surface roughness of Ra = 20 nm, without the need for polishing the window. The method allows the growth at different sizes and shapes, leaving a convenient silicon frame to protect window. The low roughness of the windows is important for improved spatial coherence of the beam. The characterization of both surface morphologies of nanocrystalline diamond windows by SEM and AFM have shown their low roughness. Raman spectroscopy analyzed film quality. The transparency and scattering tests were performed using the synchrotron beamlines at LNLS (Brazilian Synchrotron Light Laboratory). Thicker windows, of up to 40 μm, allowed pressure tests assisted by a mechanical pump in a sealed chamber.

2. Experimental

The substrates where (100) p-type silicon of 1–20 Ω cm (Wafer Worl. Inc.). The HFCVD reactor used for the growth of nanocrystalline diamond films had 6 straight tungsten filaments with diameters of 85 μm or 125 μm, equidistant by 4 mm. Filament temperature was around 2200 °C. Gas concentrations were 0.5 vol.% methane, 75 vol.% Argon and 24.5 vol.% hydrogen, at total flow rate of 100 sccm [18]. The working pressure was 6.6 kPa. Substrate temperature control between 600 and 700 °C was key factor for a good quality of the nanocrystalline film. This same reactor was also used for microcrystalline diamond (MCD) growth just by changing only the gas mixture to 2 vol.% CH_4 in H_2.

For substrate seeding by ESND the first step was to chemically attack silicon substrate with a mixture of hydrofluoric acid – BOE (Buffered Oxide Etchants) for about 40 s to remove native oxide layer [19]. The second step was to modify silicon substrate surface energy by dipping it in a solution with 10 wt% of the cationic polymer Poly diallyldimethylammonium chloride – Mw 40000 (PDDA) in DI water. The minimum immersion period was 30 min. A goniometer Krüss Easy Drop Standard using D.I. water allowed the analysis of the surface energy change. The third step was the seeding itself, in which silicon substrate removed from the PDDA solution was immersed in a nanodiamond seeding dispersion for 30 min (without agitation). After each step the silicon substrate was washed in DI water. A flow of nitrogen gas dried the seeded substrates.

The seeding dispersion preparation used diamond powder with 4 nm particle size. The 4 nm diamond powder commercially available really forms rigid clusters that can reach up to micrometer sizes. These clusters arise because of particles electrostatic interactions and covalent bonds between the surface functional groups, as well as have a soot-like structure around the primary particles [20,21]. Deagglomeration of these clusters was necessary to get the seeding dispersion. It took place inside a Teflon container under high-power sonication (750 W Sonics VCX 750). Diamond powder was mixed with DI water, zirconia (ZrO_2) spheres and the dispersing agent PSS (Poly (sodium 4-styrenesulfonate)) – as described in Table 1. Both, the friction of the diamond nanoparticles with zirconia spheres and ultrasonic cavitation help in breaking the clusters [22].

Table 1
Parameters used in deagglomerating of the 4 nm diamond particles.

Material	Parameters
D.I water	200 ml
PSS	15 g
ZrO_2 (0.4–06 mm)	250 g
Diamond 4 nm	10 g
Sonication (time)	2 h

Fig. 1. Molybdenum mask models.

After seeding the silicon substrate was inserted in the HFCVD reactor for NCD growth. Growth time controls NCD film thickness. Preparation of the window frame followed NCD growth. First, the HFCVD reactor was open and a molybdenum mask in the form of the window was placed over the NCD film. The reactor was

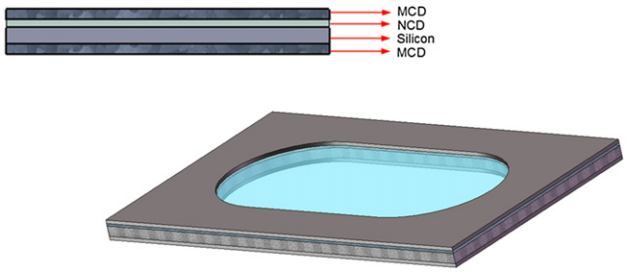

Fig. 2. Schematic representation of the frame.

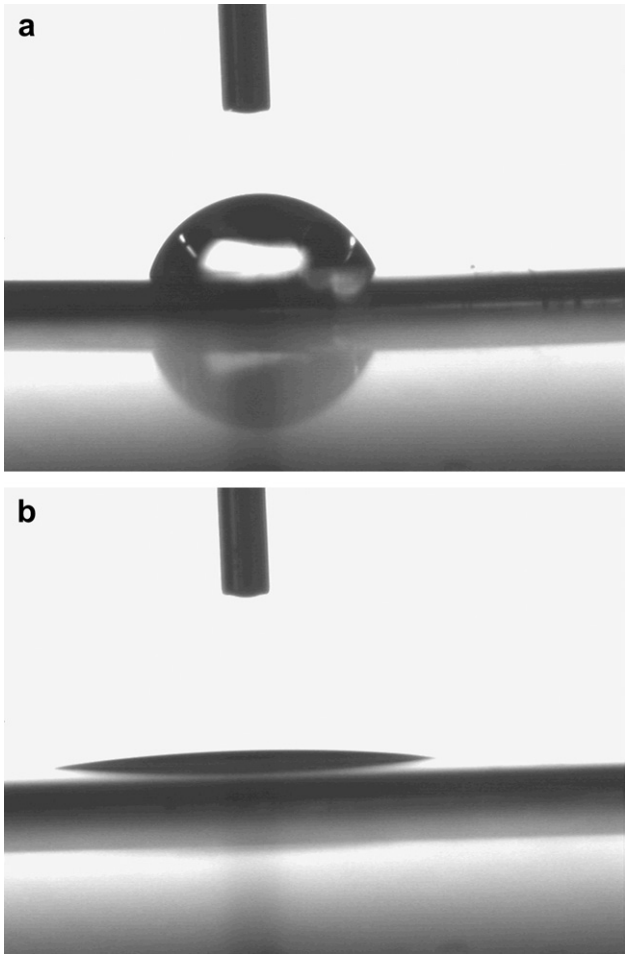

Fig. 3. Contact angle measurement: (a) – Silicon substrate without PDDA, (b) – Silicon substrate with PDDA.

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