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Diamond and Related Materials 13 (2004) 954–957
DIAMOND AND RELATED MATERIALS
www.elsevier.com/locate/diamond

Optical properties of diamond-like cladding for optical fibres

M. Smietana[a,*], J. Szmidt[a], M. Dudek[b], P. Niedzielski[b]

[a]Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Koszykowa 75, 00-665 Warsaw, Poland
[b]Institute of Materials Science and Engineering, Technical University of Lodz, Stefanowskiego 1/15, 90-924 Lodz, Poland

Abstract

This article presents a novel application of DLC layers: cladding material for silica optical fibre's core. The coats were successfully obtained using the radio frequency plasma chemical vapour deposition (RF PCVD) method. A technological subject of depositing diamond-like carbon layers onto dielectric substrates is also raised. An original way of placing samples in a plasma reactor and a process taken in order to get a desirable effect were worked out. Different values of optical fibre transmission, which depend on the RF PCVD process parameters, can be observed. Obtained coats can be characterized by good adhesion and stability. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) images of the deposited DLC layer were made. A partly uncoated optical fibre, covered by the DLC layer, can act as a sensing head. The experiments prove very high sensitivity to humidity depending on a length of the uncoated section and deposition process parameters.

Keywords: Diamond-like carbon; Plasma CVD; Optical properties characterization; Sensors

1. Introduction

Diamond-like carbon layers are well known as a passivation coating for many electronic devices [1]. DLC layers can find their application as protecting, passivating and antireflecting coats for many surfaces [2–5]. Layers of this type can be also deposited onto glasses and SiO_2 aerogels [6]. Excellent chemical inertness of a diamond-like film makes it a promising material for medical implants, cardiovascular surgery and for coating certain components of artificial heart valves [7–10]. The RF PCVD method gives an opportunity to deposit a DLC film on some polymers without damaging them significantly. During the process some advantages of the deposition method were taken such as, a low temperature process (which can be optimised depending on a concrete surface) or good DLC layers adhesion to an applied surface [11–13]. A novelty of the work is depositing a diamond-like carbon layer onto the polymer clad silica (PCS) optical fibre (OF), commonly used for optoelectronic sensors [14]. The central part of approximately 15-cm long optical fibre was prepared for layer deposition by removing polymer cladding. Measurements of this system's transmission were taken and compared with the transmission of the same system drowned in pure water. An influence of several deposition process parameters on system's transmission was checked using this method.

2. Experimental details

2.1. Optical fibre preparation

The presented experiment takes an advantage of a very popular refractometric tool based on partly uncoated optical fibre [15]. The PCS optical fibre consists of the SiO_2 core (∅ 400 μm) covered with silicone cladding and an exterior protecting polymer coat. Both layers were mechanically removed, allowing the DLC cladding deposition directly onto the SiO_2 core. The way of removing silicone cladding has to be improved because it was not removed perfectly each time. Almost a hundred of optical fibres were prepared using this method paying attention to accurate length of cladding removed section. Two types of fibre's uncoated section length were prepared: a 25-mm long series and a series of a different section length ranging from 5 to 45 mm with 5-mm intervals. All fibres were cleaned and prepared to plasma precleaning by an acetone bath.

*Corresponding author. Tel.: +48-22-625-73-29; fax: +48-22-625-73-29.
E-mail address: msmietan@elka.pw.edu.pl (M. Smietana).

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doi:10.1016/j.diamond.2003.12.003

Table 1
The main parameters for radio frequency plasma chemical vapour deposition

Substrate	SiO_2 and Si
Gas	Methane
Pressure (Pa)	25
RF generator frequency (MHz)	13.56
RF generator power (kW)	6
Negative self-bias voltage of RF powered electrode (V)	180, 240, 300, 360
Deposition time (min)	3, 5, 7, 9, 11

2.2. Plasma precleaning and DLC cladding deposition

The film was deposited by the RF decomposition of methane (RF PCVD). The experimental values are given in Table 1. The optical fibres were placed in special 'U-type' holders with keeping a 10 and 25-mm distance from a RF powered negatively self-biased electrode. Simultaneously, a piece of silicon substrate was placed on the electrode next to the mentioned holder each time. Every deposition was preceded by a 3-min long plasma etching with self-bias voltage equals to -420 V.

2.3. Measurement set-up

The tips from both sides of each optical fibre were cut off. OF was placed in a special holder ensuring system's balance during measurements. The middle section of each OF was covered by a container allowing to fill it with some liquid (H_2O, refractive index $n=1.32$). One end of the OF was supplied by a laser diode ($\lambda=670$ nm) whose amplitude was modulated with 1 MHz frequency. The other tip of the OF was connected to a detector system allowing a gain of input signal and demodulation. Four values of the transmission were measured: dry OF transmission, transmission for OF held over the liquid, transmission of OF drowned in the liquid and transmission of OF pulled out rapidly from the liquid.

2.4. DLC fibre's cladding and DLC film on Si analyses

Three types of DLC layer analyses were done: Scanning Electron Microscopy images with *Jeol JSM 5200*, Atomic Force Microscopy images with *NTMDT Solvr P47* and ellipsometric measurements with *Gaertner L116* ($\lambda=623.8$ nm). All mentioned analyses were carried out on the layers deposited onto silicon surfaces accompanying each OF. Only SEM images of protected by thin gold film OF could be done.

3. Results

Deposited DLC cladding on PCS optical fibre is solid (Fig. 1). The film deposited onto a SiO_2 substrate with the RF PCVD method can be characterised by good adhesion and durability. The SiO_2 core was significantly reinforced by depositing the DLC cladding. The film is approximately 150/300-nm thin, refractive indexes are 2.1/2.5. We can assume that the layers deposited onto Si substrates are comparable to layers deposited during the same process onto SiO_2 fibres' cores.

Two kinds of process were observed: with stable self-bias voltage during the whole process, and the other with fluctuating self-bias voltage. The rest of parameters for both processes were the same and stable. Moreover, different colours of layers deposited onto Si surfaces can be observed in both types of process. Ellipsometric measurements of layers achieved with unstable process were hardly possible. AFM researches also proving their short-term durability. Bringing up unstable type of process is caused by its frequent appearance. An unstable process course is considered as characteristic for plasma methods. The results of transmission's measurements are significantly different for both types of processes.

In Fig. 2 one can observe main differences in transmission behaviour for a stable and unstable type of process. For the stable process, rises in transmission and for unstable one, falls in it can be observed, depending on active (DLC cladding) area. Not linear dependence can be caused by an imperfect silicone cladding removal (also visible on Fig. 3). In both types of process, the cladding can be characterised as significantly moistened. Both are sensitive to hydro vapours, which can be observed by transmission changes during staying the DLC section over water surface.

Dependence on self-bias voltage transmission rises is observed for higher values (e.g. -360 V) and falls for low values (e.g. -180 V). Dependence on deposition time can be noticed as rises in transmission for short time process (e.g. 3 min) and falls for long time process (e.g. 11 min). Also, a strong influence of samples placing distance with regard to RF negative self-bias

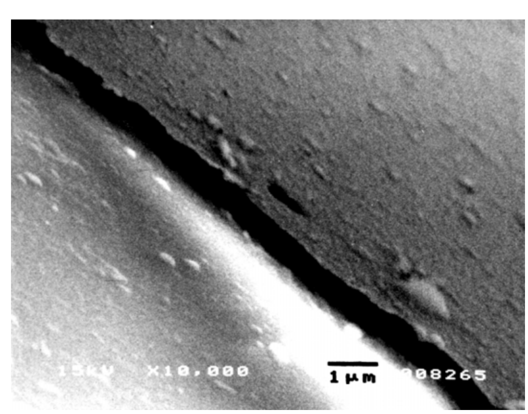

Fig. 1. SEM image of the DLC cladding breakthrough. Dark part of image is a layer and light part is a SiO_2 substrate.

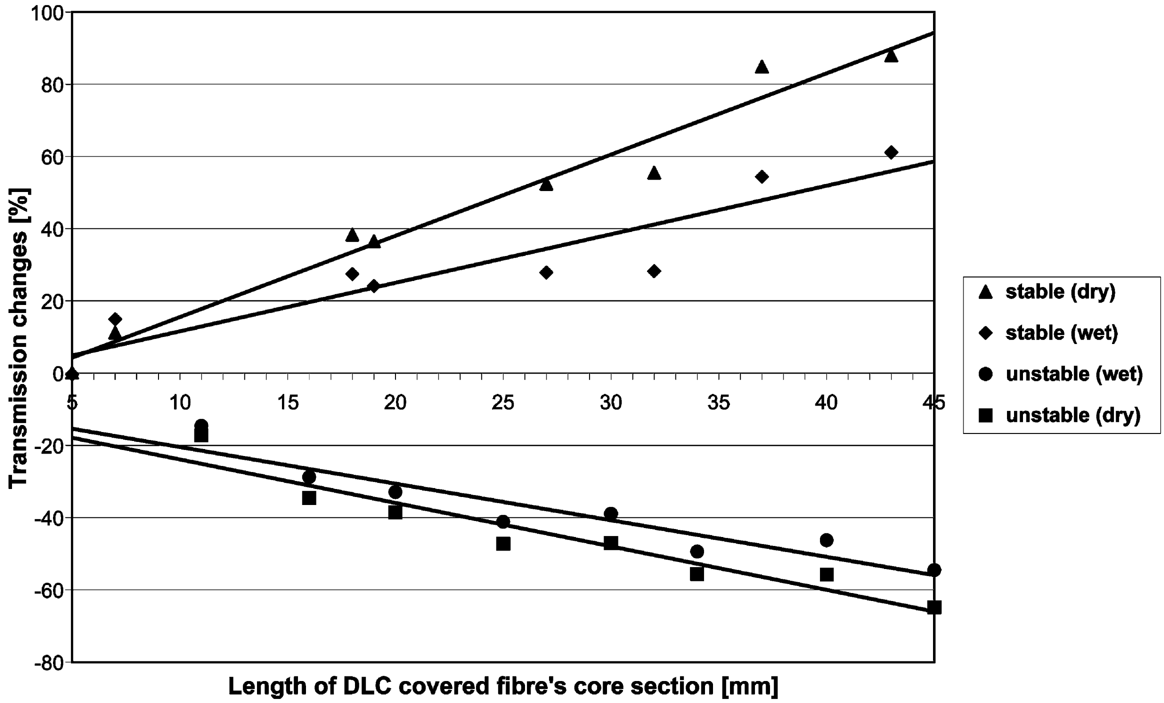

Fig. 2. The graph illustrates fibre transmission changes relative to wet and dry fibre's surface, regarding a length of DLC cover section for stable and unstable process course. (The process parameters: mounting height: 2.5 cm; self-bias voltage: −300 V; time: 5 min; working liquid's refractive index: 1,32).

voltage electrode. Placing optical fibre 10 mm over the electrode, gives high possibility of causing transmission rises. The distance of 25 mm between fibre and electrode surface is more dependent on self-bias voltage value, and one can obtain rises in transmission for higher RF voltages and falls for lower values.

In SEM images of film deposited onto optical fibres' cores common cracks can be observed. In film deposited with an unstable process cracks are wider and there are more of them, comparing with the film achieved by a stable process. In AFM images roughness of film deposited in unstable process can be observed (Fig. 4).

4. Conclusions

Unique properties of DLC such as biocompatibility and mechanical hardness, make it possible to product biomedical applications. DLC coats can protect fibre's core during its contact with a living organism. Fibre optic cores with diamond-like carbon cladding can find their practical applications as probes delivering optical signal into and out of such an organism. By adjusting certain DLC deposition process parameters we can achieve probes characterised by falls or rises of transmitted signal during a contact with wet surrounding.

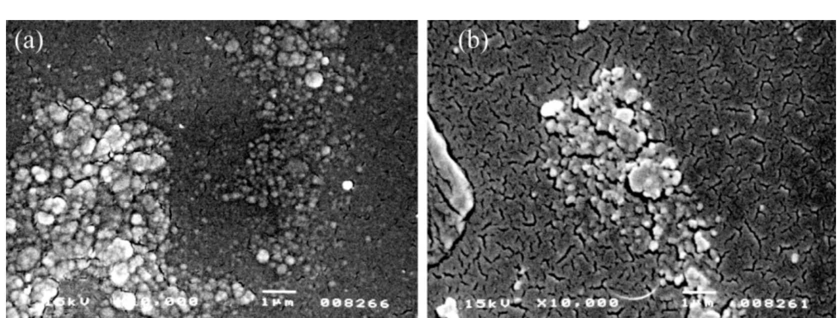

Fig. 3. SEM images of the DLC layers covering SiO_2 fibre's core for both types of transmission: rises (a) and falls (b). Process parameters the same like for Fig. 2.

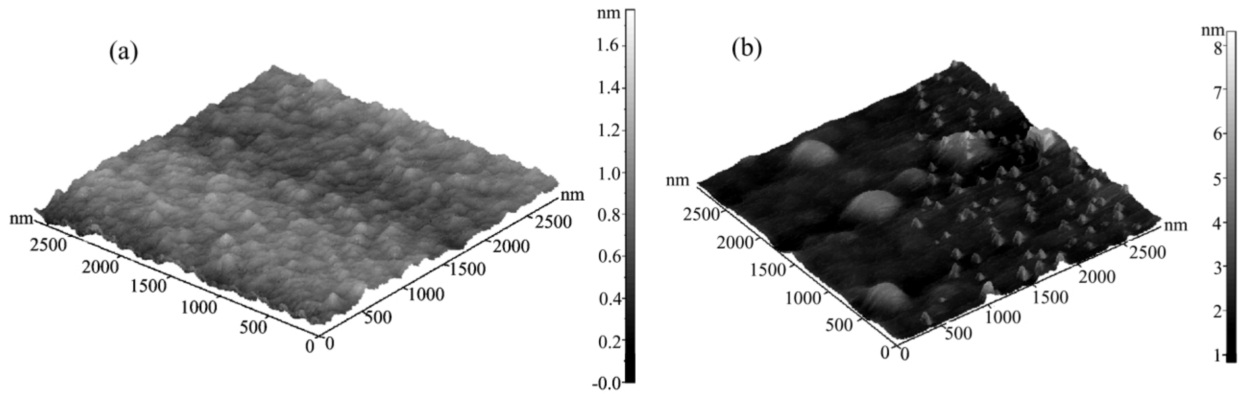

Fig. 4. AFM images of the DLC layers covering Si for both types of process: stable (a) and unstable (b).

Such probes can also act as deposition process appropriate course detectors. The presented method of process diagnosis is cheap and easy to put into practice. The presented probes can also act as humidity detectors. Works on diamond-like carbon cladding for optical fibre will definitely be carried on further.

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