Microcrystalline diamond film evaluation by spectroscopic optical coherence tomography

Paulina Strąkowska,[*1] Marcin Strąkowski,[2]

[1]*Faculty of Mechanical Engineering and Ship Technology, Gdańsk University of Technology, Narutowicza 11/12, 80-233 Gdańsk,*
[2]*Faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology, Narutowicza 11/12, 80-233 Gdańsk*

Received May 23, 2022; accepted September 08, 2022; published September 30, 2022

Abstract—This study has focused on microcrystalline diamond film (MCD) thickness evaluation. For this purpose, optical coherence tomography (OCT) enhanced by spectroscopic analysis has been used as a method of choice. The average thickness of the tested layer was about 1.5μm, which is below the standard 2-point OCT resolution. In this case, the usefulness of the spectroscopic analysis was confirmed for the evaluation of thickness changes in the submicrometer range.

Diamond thin films and coatings are of interest in many areas including science, medical and industrial applications. Due to their unique features, they can be utilized in a wide spread of applications from mechanical engineering by the semiconductor industry to medical components and implants [1–4]. The main object of this research is the microcrystalline diamond film designed for implant coating [5]. One of the key factors in film development is to obtain its continuous structure with uniform thickness. For this purpose, optical coherence tomography with spectroscopic analysis has been used as a method of choice.

Optical coherence tomography (OCT) is an imaging technique, which uses low-coherent light to acquire the 2D and 3D tomographic images of scattering and semitransparent objects. The images are captured with micrometer resolution depending on the optical scanning head and the spectral characteristic of the scanning light beam [6–7]. The metrological abilities and the standard OCT limitations make this technique applicable for microscopic evaluation, especially in medical diagnosis [8–9]. The advantage of using OCT is the possibility to investigate the devices with complex structure, e.g. composed of multiple layers, which is difficult with the aid of other methods like reflectometry or ellipsometry. In this study, the standard OCT method has been enhanced by the time-frequency analysis, which is known as spectroscopic OCT (Sc-OCT). This method is based on retrieving the backscattered signal from particular scanning depths and performing spectral analysis using time-frequency signal processing methods. As a result, spectral information about backscattered light can be gathered from particular points inside the evaluated object. Among others, Sc-OCT has been used for components recognition [6], oxygen saturation of blood [8], or the contrast agents detection inside tissues [9]. Here, the benefits of spectroscopic (time-frequency) analysis make the thin-film evaluation possible [10–11].

* E-mail: paulina.strakowska@pg.edu.pl

The chemicals used for the experiments and analysis described below were of analytical purity and were obtained from Sigma-Aldrich (Germany) unless otherwise stated.

Cylindrical specimens were made of Ti6Al4V (ASTM 136) alloy – the dimensions: the diameter is 16 mm and the height is 2 mm. The surface of the sample was grounded and polished in the final stage using a diamond suspension with a grain size of 1–3 μm. Before film deposition, the sample was sonicated in 1% Triton X-100 (Sigma-Aldrich, Germany) in deionized water, acetone, and ethanol for 20 minutes each.

The first stage of the deposition process of diamond layers was nucleation using a nanodiamond suspension based on DMSO dimethylsulfoxide (Blueseeds, ITC, USA) by immersion or sonication for 1 hour in an ultrasonic cleaner (ELMA S40H Elmasonic, Germany). The suspension mass concentration was 0.5%.. Before placing the samples in the processing chamber, they were dried with a stream of nitrogen. Deposition of MCD layers was carried out using the MWPA-CVD method (Microwave Plasma Assisted Chemical Vapor Deposition, Astex AX6500, Japan) using a mixture of methane and hydrogen gases in a percentage ratio of 1:99. A process pressure of 50 Torr and a total flow rate of 300 sccm were used. The power of microwave (PMW) used to excite the plasma was kept at 1.3 kW. A layer containing microcrystalline diamond (MCD / TiC) was obtained. The deposition time of the layers was 180 min and the substrate temperature (TS) was 700 °C.

The obtained microcrystalline diamond layer was tested for structure and topography. For this purpose, three

methods were used: atomic force microscopy (AFM), the scanning electron microscope (SEM) and the Ramman Spectroscopy. With the use of AFM, the necessary information on the topography and surface roughness of the tested material has been presented in Fig. 1. For this purpose, a microscope from N-TEGRA Prima, NT-MDT Company, Moscow, Russia was used. Microstructural studies of the morphology of the obtained surfaces were performed on a Philips XL13ESEM scanning electron microscope in the range of low accelerating voltage (3 kV) after the surface was covered with a thin layer of carbon (Baltec, Germany).

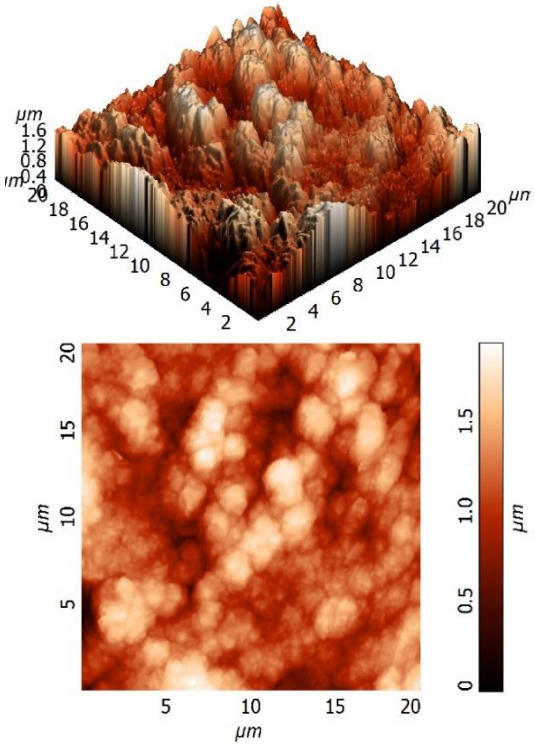

Fig. 1. The MCD layer topography obtained by the AFM.

The resulting SEM image (Fig. 2) shows that the MCD layer consists of two structures: TiC and 1–2 μm diamond crystallites formed on it, covering about 5% of the surface. In some places, diamonds ware arranged in a form of groups of crystallites of similar size. It is not possible for a non-invasive test to determine the thickness of deposited layer because of the heterogeneous structures. The parameters describing the surface roughness of the deposited layer are the average roughness (Ra) and the root-mean-square roughness (Rq) – measured by the AFM. The coating was scanned on an area equal to 20×20 μm^2 with an AFM HA-NC (High Accuracy NonContact) probe (NT-MDT, Moscow, Russia) operating in a semi-contact mode with a resonant frequency f_r = 261.189 kHz.

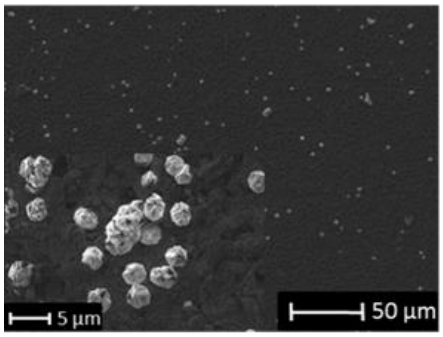

Fig. 2. The SEM image of the MCD layer [5].

The MCD layers were deposited on a substrate made of a Ti6Al4V titanium alloy, previously properly prepared. The deposited coating was identified using a JOBIN-YVONE T64000 Raman microscope. Bands of 391 cm^{-1} and 612 cm^{-1} were observed on the surface of the deposited layer, which means the presence of a competitive titanium carbide (TiC) phase surrounded by an amorphous carbon phase.

The measurement method of choice was optical coherence tomography with spectroscopic analysis (Sc-OCT). The OCT is capable to provide the spatially resolved detection of back-scattered light from particle points inside the tested device. Following, the single depth-resolved scanning line, called A-scan, depends on the positions (in-depth) of the light scattering or back-reflecting components as well as their spectral characteristics. To explain the usefulness of spectroscopic analysis, it is necessary to refer to the thin film reflectance phenomena. In general, the back-scattered light from a semitransparent thin film is a product of the Fresnel principles and the interference of multi-scattering beams inside the film. In this case, the spectral characteristic of the back-scattered light may vary referring to the film thickness, which might be detected and analysed by the OCT enhanced by spectroscopic analysis [4]. Due to the periodic function of the film reflectivity, the exact film thickness not always can be estimated. However, its variation may be observed with submicrometer resolution. This has been achieved by monitoring the shift of local minima at the spectral characteristic of backreferenced light, delivered by the spectroscopic analysis in the OCT.

The spectroscopic analysis has been added to the standard OCT system. This Sc-OCT system is running under customized software, designed and developed at the Gdańsk University of Technology. The main features of the standard OCT system were summarized in Table 1.

Table 1. Main features of the OCT system.

Features	Values
Light source type	20 kHz swept-source
Aver. output power	10 mW
Central wavelength	1290 nm
Wavelength range	140 nm
Axial resolution	12 μm (in the air)
Lateral resolution	15 μm

The MCD sample was scanned to obtain the C-scan OCT image – the en-face plane. The scan covered the 5 mm by 5 mm area at the centre of the tested sample. For each point of the C-scan, the spectral characteristic of back-scattered light was obtained and the changes in the film thickness were estimated. The obtained results by the Sc-OCT system were presented in Fig. 3 as a map of thickness changes of the tested MCD layer.

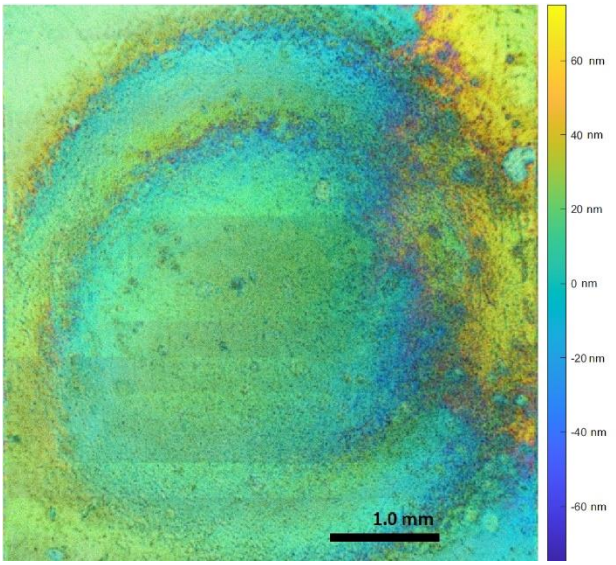

Fig. 3. The map of the MCD sample thickness changes obtained with the aid of the OCT enhanced by spectroscopic analysis.

The applied spectroscopic analysis delivers the spectral characteristic of the backscattered light from particular points inside the tested sample. The number of analyzed points was limited only to those related to the MCD thin film. The analysis was performed by observing the shift of the local minima of the backscattered light spectra [10]. Its position in the spectral domain has been compared with the spectral reflectance characteristic of the layer, calculated on the basis of the AFM and SEM measurements results. Afterwards, the map of the film thickness changes was obtained and presented in Fig. 3. This is a wrapped function, which means that two green points (marked as 0 nm) separated by yellow and violet areas differ about 180 nm in elevation.

This study shows the benefits of using spectroscopic analysis in combination with OCT. The thickness changes can be observed with a fine accuracy on the submicrometer scale. The measurements can be made very fast, which is one of the advantages of the OCT method. The evaluated film can be covered by another semi-transparent layer, which was presented in [10]. However, there are some limitations of this approach, which needs to be underlined. Due to the periodic character of the film reflectance, it is difficult to estimate the exact thickness. This problem becomes crucial for thin layers, where only one local minimum in the spectral range of a swept light source is visible. Therefore, the application of the Sc-OCT has been limited to tracking changes in the layer thickness.

This research was funded by the DS programs of the Faculty of Electronics, Telecommunications and Informatics, and the Faculty of Mechanical Engineering and Ship Technology, Gdańsk University of Technology.

References

[1] M.D. Drory, J.W. Hutchinson, Science **263**, 1753 (1994).
[2] J. Wang, J. Zhou, H.Y. Long, Y.N. Xie, X.W. Zhang, H. Luo, Z.J. Deng, Q. Wei, Z.M. Yu, J. Zhang, Z.G. Tang, Surf. Coat. Technol. **258**, 1032 (2014).
[3] P.A. Nistor, P.W. May, F. Tamagnini, A.D. Randall, M.A. Caldwell, Biomaterials **61**, 139 (2015).
[4] C.A. Love, R.B. Cook, T.J. Harvey, P.A. Dearnley, R.J.K. Wood, Tribol. Int. **63**, 141 (2013).
[5] P. Strąkowska, R. Beutner, M. Gnyba, A. Zielinski, D. Scharnweber, Materials Science and Engineering: C **59**, 624 (2016).
[6] T.S. Ho, P. Yeh, C.C. Tsai, K.Y. Hsu, S.L. Huang, Opt. Expr. **22**, 5 (2014).
[7] N. Bosschaart, T.G. van Leeuwen, M.C.G. Aalders, D.J. Faber, Biomedical Opt. Expr. **4**, 11 (2013).
[8] F.E Robles, C. Wilson, G. Grant, A. Wax, Nature Photonics **5**, 12 (2011).
[9] A.F. Fercher, W. Drexler, C.K. Hitzenberger, T. Lasser, Reports on Progress in Physics **66**, 239 (2003).
[10] A.M. Kamińska, M.R. Strąkowski, J. Pluciński, Sensors **20**, 19 (2020).
[11] M. Kraszewski, M. Strąkowski, J. Pluciński, B.B. Kosmowski, App. Opt. **54**, 1 (2015).

