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REVIEW ARTICLE

DIAMOND-LIKE CARBON FILMS: IMPROVED PROPERTIES AND NEW APPLICATIONS

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ABSTRACT

Since 1972, diamond-like carbon (DLC) thin films have attracted great scientific and technological interest due to their unique properties, notably the high adhesion on metallic and non-metallic substrates, allowing them to expand their applications to areas such as space and biological. DLC films can be obtained by various techniques, such as physical vapor deposition, ion beam-assisted deposition, and plasma enhancement chemical vapor deposition (PECVD). This review focused on the studies of the Diamond and Related Materials Research Group, which compared various techniques and selected direct current (DC) pulsed PECVD for its low cost and versatility. The studies focused on determining the coating parameters such as adhesion, hardness, friction, wear resistance, biocompatibility, structural stresses, and scalability. Next, the group improved the DC pulsed PECVD with the introduction of the concept of ion and electron confinement. This innovative method has made it possible to obtain better plasma density at low pressures, improving the characteristics of the film. The non-collision growth process resulted in a harder DLC, with better adhesion, less wear, and maintained biocompatibility. The system has proven to be effective, cost-effective, easy to use, and scalability even for complex geometries.

KEYWORDS: DLC films, Growth process, PECVD techniques, Properties, Applications.

FILMES DE CARBONO TIPO DIAMANTE: PROPRIEDADES APRIMORADAS E NOVAS APLICAÇÕES

RESUMO

Os filmes finos de carbono tipo diamante (DLC), desde 1972, atraem grande interesse científico e tecnológico por causa das suas propriedades únicas, notavelmente a alta aderência em substratos metálicos e não metálicos, permitindo expandir suas aplicações para áreas como espacial e biológica. Os filmes de DLC podem ser obtidos por diversas técnicas, como deposição física na fase vapor, deposição assistida por feixe de íons e deposição química na fase vapor aumentada por plasma (PECVD). Esta revisão focou nos estudos do Grupo de Pesquisa de Diamantes e Materiais Relacionados, que comparou várias técnicas e selecionou a PECVD de pulso de corrente contínua por seu baixo custo e versatilidade. Os estudos concentraram-se na determinação dos parâmetros de revestimento, como adesão, dureza, fricção, resistência ao desgaste, biocompatibilidade, tensões estruturais e escalabilidade. Em seguida, o grupo aprimorou a PECVD de pulso de corrente contínua com a introdução do conceito de confinamento de íons e elétrons. Este método inovador permitiu obter melhor densidade de plasma a baixas pressões melhorando as características do filme. O processo de crescimento em regime de não colisão resultou em um DLC mais duro, com melhor adesão, menor desgaste e biocompatibilidade mantida. O sistema demonstrou-se eficaz, econômico, de fácil uso e de escalabilidade mesmo para geometrias complexas.

PALAVRAS-CHAVE: Filmes de DLC, Processo de crescimento, Técnicas PECVD, Propriedades, Aplicações.

INTRODUCTION

Due to the wide scope of the area of studies in diamond-like carbon (DLC) films from a scientific and technological point of view and its wide range of applications, the community elect itself as the main coating to be investigated. About 25 years ago, a complete review emphasizing all deposition techniques was published[1]. Nowadays, there are several recent review articles segmented by area of study and/or area of application[2–4]. Therefore, this review focused on the works developed in the DLC research related to fundamental and application studies project. Some comparative approaches to other research available in the literature were done.

Initially, several growth techniques were studied. Among them, physical vapor deposition (PVD) itself with magnetron sputtering, which was related to cold welding prevention[5]. These results were not satisfactory due to the low adhesion on the different substrates, like titanium and aluminum, but they were the first results related to space applications from the Diamond and Related Materials Research Group.

The initial results of the radiofrequency plasma enhanced chemical vapor deposition (PECVD) technique showed great difficult in obtaining good adhesion and in scaling due to the impossibility of manufacturing high power radiofrequency sources to reach large deposition areas[6,7]. Besides this technique, two other techniques were studied in the team at the same time. First, the ions beam assisted deposition (IBAD)[8,9]. It was observed that the adhesion in relation to the radiofrequency-PECVD technique improved, but it was a relatively expensive system, difficult to operate and very limited in terms of deposition area, as well as in terms of deposition in three dimensions. Despite some good results in terms of adhesion, efforts were concentrated on the pulsed direct current (DC) PECVD technique[10–16]. This technique was under study by several research groups around the world[17,18].

The study examined various growth factors, including pulse width, frequency, and amplitude, in relation to the gas pressure within the discharge. It also considered both the internal and external stresses created on the film, alongside the uniformity of growth across larger surfaces and the three-dimensional aspects of the deposition process.

Some prominent publications showed that the working pressure varied greatly, from 1–6 Pa for pulse frequencies of 2.3 MHz, to 200 Pa[17] for frequencies of 25 to 200 kHz[18]. It was also shown that it was difficult to obtain good adhesion; the internal and external stresses of the film were relatively high, even in small thicknesses. Also, it was observed that the growth temperature to obtain good quality and reasonable adhesion was very high. The pulsed DC PECVD technique is the one that promised to be most suitable for studying the dependence of the growth parameters with the properties of the film, and at the same time can be obtained at a lower cost and with friendlier handling.

Furthermore, advancements have been made in methods like immerson ion implantation plasma deposition (IIIPD), although challenges persist in achieving adequate adhesion and scale over large surfaces. These challenges stem from the high voltages needed, ranging from 20 to 50 kV, and the slow growth rates associated with various parameters[19–21].

Another prominent technique for DLC growth is high power impulse magnetron sputtering (HIPIMS)[22], which is a variation of the PVD technique and has greatly improved adhesion when additional surface treatments are incorporated. On the other hand, it is a more expensive technique, and the issue of growth in three dimensions remains without a good solution.

As a result, the team's primary goal was to thoroughly research focused on the pulsed DC PECVD technique. Many additional works were published by the team, involving mechanical, tribological, chemical, and biological properties, covering all industrial segments, including the space area, in which an ultra-high-vacuum environment is required. All segments are treated separately, intending to show the intense research, development, and innovation activity during and after the introduction of the important modification in the deposition system. As one can see, DLC films have been improved not only in terms of different procedures, but also concerning relevant modifications using a simple physical concept.

PULSED DIRECT CURRENT PLASMA ENHANCED CHEMICAL VAPOR DEPOSITION TECHNIQUE CONCEPT

Directed studies related to pulsed DC PECVD led us to upgrade the technique. It allowed us to develop an ion and electron confinement system that provided substantial improvements in the DLC film properties. The principle used to ensure the low working pressure that allowed DLC growth at low and high voltages in a non-collision regime (low pressure) became very important, because it allowed a better use of the energy of the ions in the growth process. Due to the absence of collisions, the average free path increases, allowing the precursor ions of the growth of the DLC film to reach the substrate surface with higher energy, promoting better adhesion and higher density. So, one can add the best of the IBAD technique, which is the adhesion, and the best of the pulsed DC PECVD technique, that is the variation of pressure, flow, and composition of the gases and the voltage used in the growth process. In the first case, besides the improvement in adhesion, one can consider a high gain in the three-dimensionality and uniformity of the deposition. In the second case, it allowed power supply technology to be possible even in large deposition areas, allowing scaling up, even at voltages as high as -15 kV, as the current is relatively low. The pulse rate ranged from 20 to 70 kHz for this study.

As shown in Fig. 1, the pulsed DC PECVD system can be electrically powered by different waveform configurations[13], and it was possible to obtain optimizations regarding pulse width, amplitude, and frequency. By varying the widths, a, b, c, and d, and the amplitudes *Vba* and *Vbc*, it was possible to find the best values of hardness, coefficient of friction, and stress for different applications, *i.e.*, for different substrate materials.

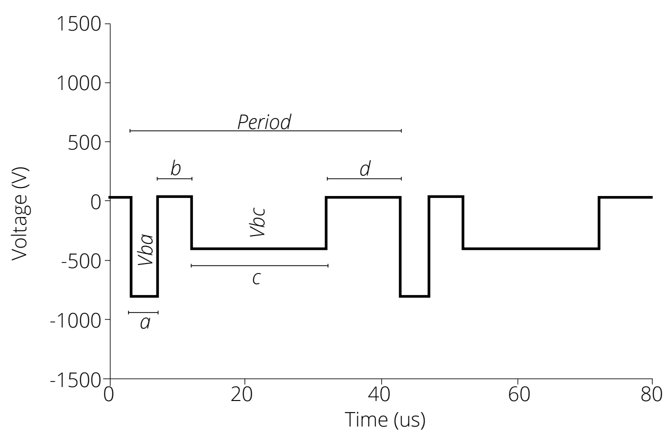

Figure 1: Voltage signal waveform driven to the cathode by the pulsed direct current source in methane discharge. Two negative–positive pairs of pulses represent one period. V_{ba} and V_{bc} are the variable negative voltage amplitudes. The time for each pulse is indicated by *a, b, c*, and *d*.

Source: Trava-Airoldi et al.[13].

The best results were for null V_{bc}, V_{ba} with a negative component (for ion acceleration), and a small positive component to help remove positive charge accumulation from the substrate surface, with a pulse rate of 20 to 30 kHz, and the duty cycle—defined as a/(a+b+c+d)—ranging from 5 to 40%. More specifically, duty cycle corresponds to the ratio of the time of the pulse on and the total period of the pulse. This parameter is very important, because it is related to the ion lifetime.

Through this system, it was possible to study the introduction, for the first time, of nanoparticles of metals, titanium dioxide, and diamond during the growth of the DLC film, giving it new properties, including for space applications[13–16], among other applications that are described later.

These studies resulted in a low-cost system[10,11], easy operation, and real possibility of scaling up, thus allowing more in-depth studies that led us to its improvement. The choice of pulsed DC PECVD technique allows us to introduce the concept of ion and electron confinement as a very important part of these studies. First, it was possible to obtain the best surface preparation to reach the best adhesion[23–35]. Adhesion is a crucial parameter for most applications.

For example, space applications were studied, both in the area of corrosion of DLC film in space environments, in which ionic oxygen in the atmosphere is highly corrosive[36,37], as well as studies to obtain low coefficients of friction in ultra-high-vacuum environment[16,38]. Also, DLC films have been studied as a component of a hybrid lubricant, seeking coefficient of friction optimization in systems that use liquid lubricants[39–41]. With the adhesion of DLC film to the key metals and their alloys, such as steels and titanium, studies more directed towards the modification of DLC film were made with the purpose of decreasing the coefficient of friction and increasing its durability with less wear. These works were best achieved by incorporating nanoparticles, such as silver (AgNP) and carbon nanotubes[15,42,43].

DLC films obtained via pulsed DC PECVD are hydrogenated, with concentrations that can vary from 20 to 50% depending on the growth parameters for a given application[16,27,30]. The hydrogenation of DLC films was also studied using photoacoustic spectroscopy and thermal diffusivity, aiming at applications such as acoustic sensors[44]. So, pulsed DC PECVD was studied as the most promising technique for DLC growth addressing improvement that will be explained as follow.

CONCEPT OF ION AND ELECTRON CONFINEMENT ON PULSED DIRECT CURRENT PLASMA ENHANCED CHEMICAL VAPOR DEPOSITION SYSTEM

Ion confinement is due to the introduction of an additional cathode, composed of a metal screen with well-defined transparency that allows a stable discharge at low pressures, about 10 times lower than in the conventional pulsed DC PECVD system. The transparency of the wire mesh, as well as its geometry and dimensions, must be optimized according to the dimensions of the discharge chamber and the one of the additional cathodes. So, geometry, dimension, and low pressure must be calculated to avoid the hollow cathode discharge limit, while maintaining the high density of ions and electrons.

Due to the difficulty of initiating the discharge, an ignition procedure was also developed, which involves a small auxiliary high-voltage electrical pulse in the presence of argon, also at low pressure (~5×10^{-4} Torr). This procedure helps to keep the plasma bound and has excellent stability, even in the presence of other gaseous components, such as N_2, H_2, CH_4, C_2H_2, etc. This concept has been studied due to the excellent initial results. All the advances related to the improvement of its physical, chemical, biological, and tribological properties are presented here[45–67]. In Fig. 2, a schematic diagram of the system and the actual discharge in a chamber with the additional cathode in working condition as an ion and electron confinement system is shown. In Fig. 3, the approximate difference in energy of the ions is shown, which form the plasma, when subjected to acceleration towards the substrate.

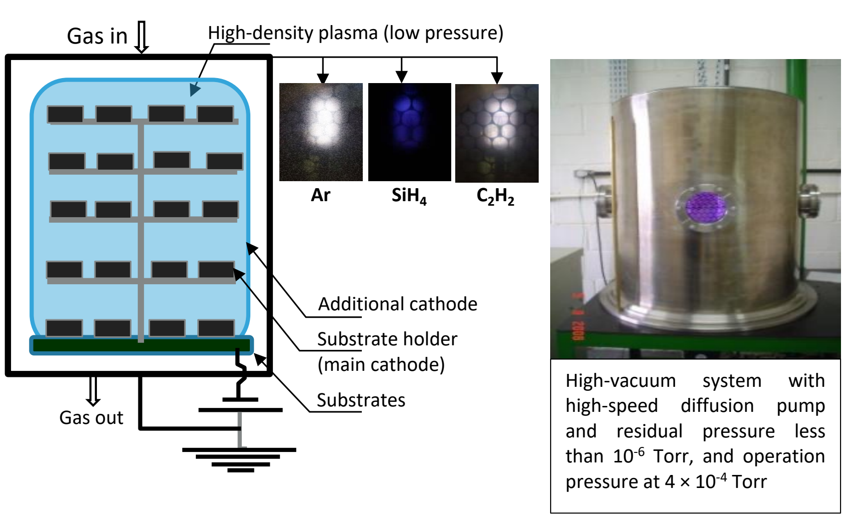

Figure 2: Schematic diagram of the system with ion and electron confinement and the actual discharge in a diamond-like carbon growth chamber.

Source: Bonetti LF et al.[45].

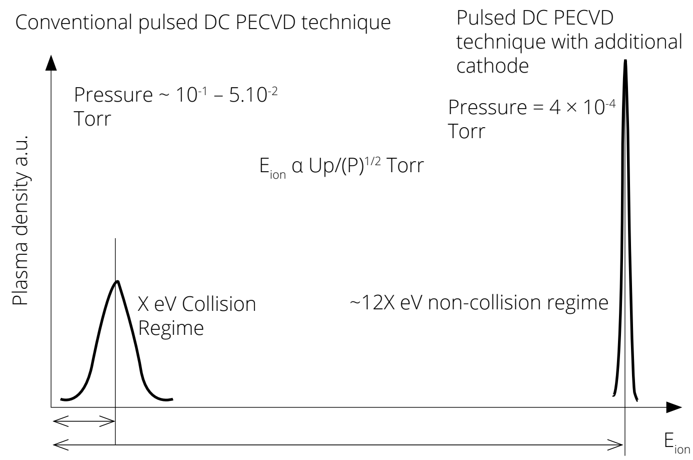

Figure 3: Difference in ion energy in a conventional pulsed direct current plasma enhanced chemical vapor deposition technique and a technique with ion and electron confinement.

Bonetti LF et al.[65].

Using this principle, it was possible to grow DLC films at low temperature (< 100°C) with growth rate control—equivalent to the conventional pulsed DC PECVD technique, quality control, lower coefficient of friction, higher hardness (lower hydrogen concentration), greater adhesion in different materials (metallic and non-metallic), lower stress, and better uniformity in the three dimensions and with well-defined scale parameters.

The following are some relevant results that led to the definitive adoption of this technique for studies in a wide range of applications.

QUALITY OF THE DIAMOND-LIKE CARBON FILM

The structural quality of DLC is evaluated by Raman spectroscopy, as shown in Fig. 4.

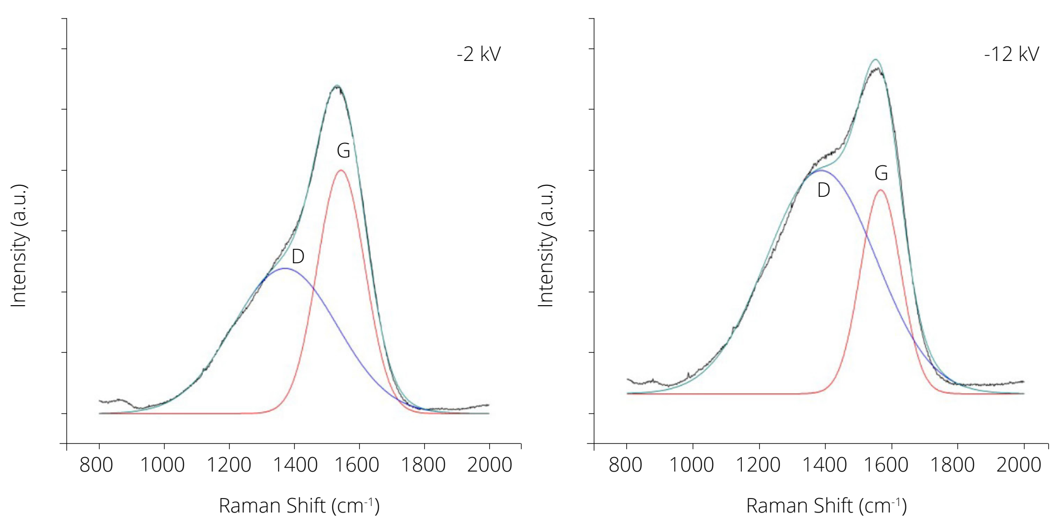

Figure 4: Two examples of Raman analysis for diamond-like carbon films deposited at -2 kV and -12 kV. The deconvolution curves show the D and G bands.

Source: Bonetti and Trava-Airoldi[45].

Figure 5 shows the variation of the structural properties as a function of the supply voltage, showing that with this technique it is possible to vary the properties of the DLC film according to the desired application.

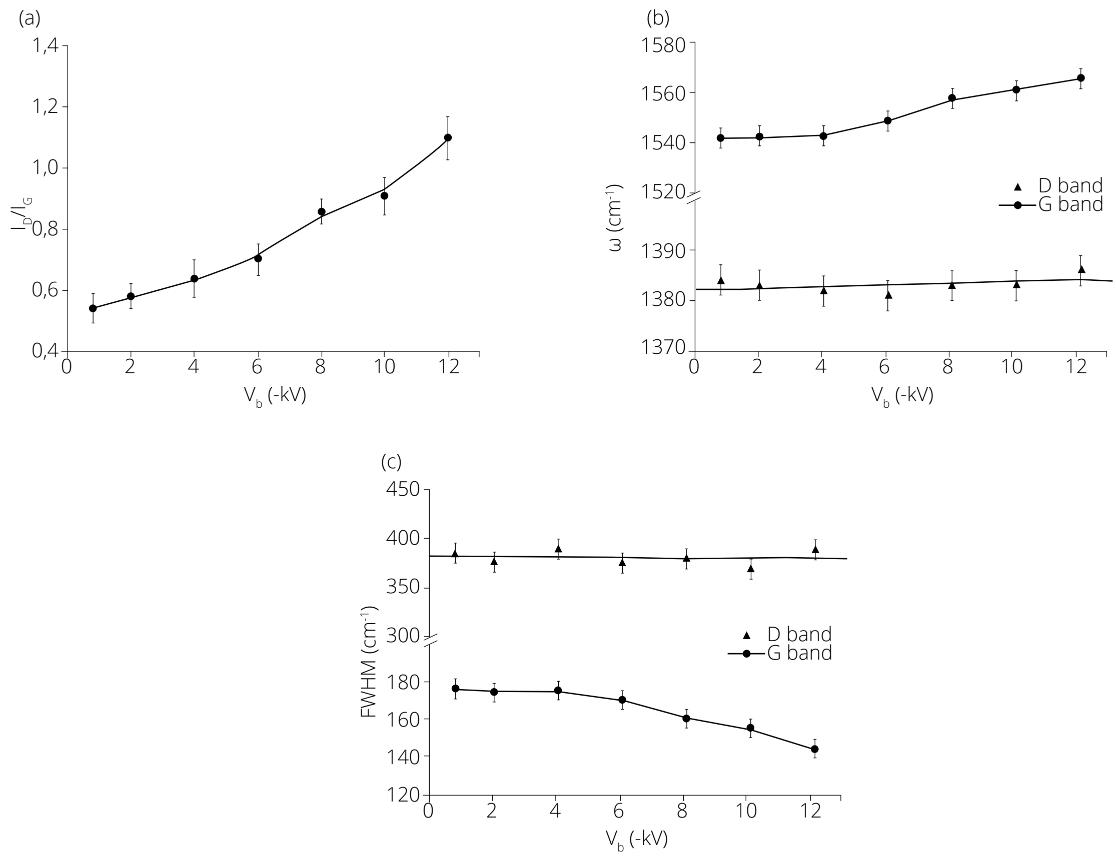

Figure 5: Properties of the diamond-like carbon film as a function of applied voltage. (a) I_D/I_G intensity ratio, (b) the band peak position (ω) of the D and G bands, and (c) the full width at half maximum (FWHM) of the D and G bands as a function of the direct current bias voltages.

Source: Bonetti and Trava-Airoldi[45].

We can observe that, according to the I_D/I_G ratio, the best condition is for lower voltage values, as well as for full width at half maximum and the positions of the D and G bands. Also, it has been observed that the concentration of hydrogen decreases with the decrease of voltage, being about 13 to 15% for voltage from -700 to -800 V, while the hardness increases with the decrease of voltage[45,46].

MECHANICAL AND TRIBOLOGICAL PROPERTIES

Mechanical properties such as hardness, elastic modulus, including adhesion measured from the Rockwell C 1,500 N indentation technique, the coefficient of friction, and wear have been intensively studied, especially to characterize the behavior of DLC on different types of materials.

The hardness and elastic modulus, as well as the coefficient of friction and wear, do not depend on the type of substrate, but only on the parameters used for the growth of the DLC film[45–58]. Because of the high energy ion bombardment, DLC films are denser than that obtained from the conventional pulsed DC PECVD technique, and some interesting phenomena have been observed, such as local transformations when subjected to strong contact pressure[52]. Still in this line of studies, the pulsed DC PECVD system with additional cathode has contributed to promote modifications of the structure of the DLC films, allowing the incorporation of different nanoparticles and doping with other elements[59–62]. On the other hand, adhesion depends heavily on the conditions of thin film deposition to form an interface, as carbon film, in general, is not very friendly to most materials, especially metallic ones, causing low adhesion.

In these studies, silane gas has been used, and voltage and pressure during the beginning of film formation, in addition to a good chemical and physical cleanliness of the substrate surface, have been well determined. Also, the formation of a silicon film gradient with DLC, overlapping it other, has a strong contribution to obtain good adhesion[45,53,55,56,65].

Different substrate materials have been used, such as Inconel alloy 718[47,65], and steel alloys without any surface treatment[48,63] and with surface treatment such as nitriding[48,51], which has also been investigated for corrosion resistance[48,55,58,63] and wear when subjected to strong pressure with counter-body of different materials[51].

Concerning different results for each type of material, comparative studies were carried out for different substrates using the same growth parameters[45,53,54]. Titanium and its alloys have been widely studied by the team, due to their various applications, especially in the space and biological areas4[5,50,59,60,61,65,68]. The adhesion of DLC in aluminum was also studied with good results, greatly expanding the applications, especially in the space, aeronautical, and biological areas. Different sets of parameters for the initial deposition of the silicon interlayer on aluminum were studied for the pulsed DC PECVD system with ion and electron confinement[65,66].

For the space, aeronautics, and biological areas, some results are presented separately here due to the novelty and scope of the applications and the use of all the improved properties of the DLC obtained by the pulsed DC PECVD technique with additional cathode. Two new applications, with DLC as a substrate for surface-enhanced Raman spectroscopy (SERS) and DLC deposition inside of long tubes, are also summarized separately.

Within the mechanical and tribological properties, the deposition of multi-layer DLC films[45,55,58,63,65,66] with their high adhesion on aluminum and composite materials[65,66] will be shown.

MULTILAYER OF DIAMOND-LIKE CARBON FILMS

Due to the possibility of controlling the growth parameters of DLC films according to the properties required for a given application and advanced requirements, the obtaining of thicker films was studied. In thicker films, the stress is very high, not allowing thicknesses beyond 2 micrometers to be obtained. Thicker films were possible with this technique, growing into multilayers separated by an interface. This interface can be achieved by the deposition of silicon concomitantly with DLC deposition or by varying the property of the DLC for a short period of time, causing the DLC film to lose the stress memory initially acquired[65]. Figure 6 shows DLC film with 10 layers and interlayers of silicon. Each layer is about 1 micrometer thick, totaling about 10 micrometers of final thickness.

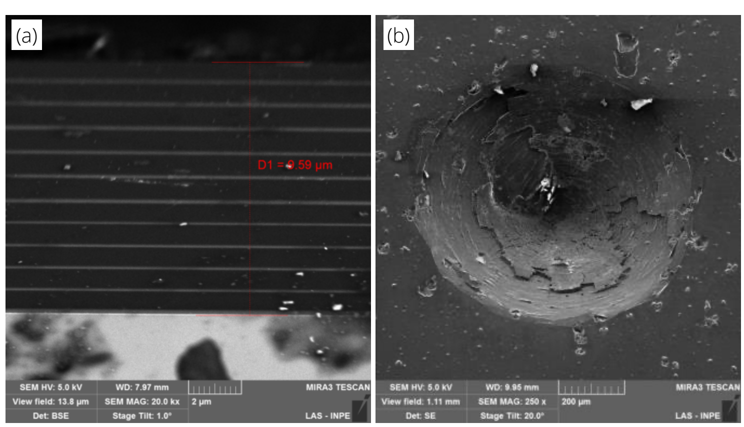

Figure 6: Rockwell 1,500 N indentation test. (a) Multilayers of diamond-like carbon with silicon interlayers on Ti6V4Al alloy and (b) indentation of 1,500 N Rockwell C.

Source: Bonetti and Trava-Airoldi[65].

It is observed that there was no delamination of the DLC film from the titanium alloy substrate. Figure 7 shows the stress formed in the DLC film as a function of the number of layers.

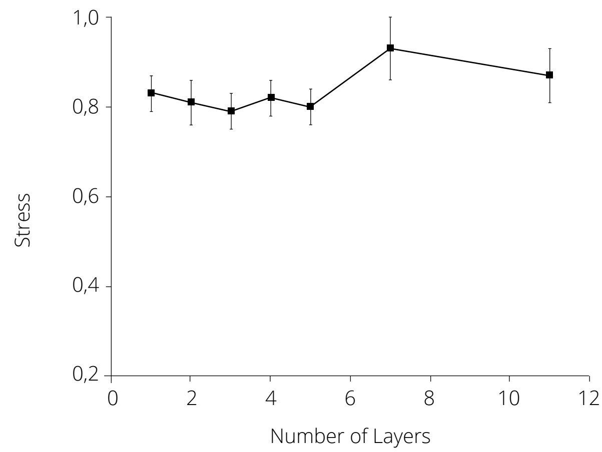

Figure 7: Stress of multilayers as a function of the number of layers.

Source: Bonetti and Trava-Airoldi[65].

It is observed that stress does not increase with the number of layers, which provides an increase in thickness without delamination, expanding the possible applications. Also, the coefficient of friction in each layer does not change, remaining approximately constant at 0.08 for all layers[65].

SPACE AND AERONAUTICAL APPLICATIONS

For space applications, the most relevant properties are the adhesion of the DLC film on the substrate surface that is independent of the material, the coefficient of friction and wear that depends on the ultra-high-vacuum environment[50,53,60,65].

Conventional techniques result in very high friction coefficient of DLC film in an ultra-high-vacuum environment. The solution with these techniques is to greatly increase the hydrogen concentration in its structure, which causes, however, a decrease in hardness and increased wear.

Pulsed DC PECVD technique with an additional cathode conversely resulted in very low coefficients of friction in an ultra-high-vacuum environment without impairing hardness and wear. Incorporating silver nanoparticles (AgNPs) and TiO_2NPs reduced the coefficient of friction in an atmospheric environment from 0.05 to 0.03[67]. Incorporation graphene nanoparticles reduced the coefficient of friction to 0.017[60], as shown in Fig. 8, keeping the wear rate constant, the hardness high, above 20 GPa, and excellent flexibility with the hardness/elastic modulus ratio above 0.13[65].

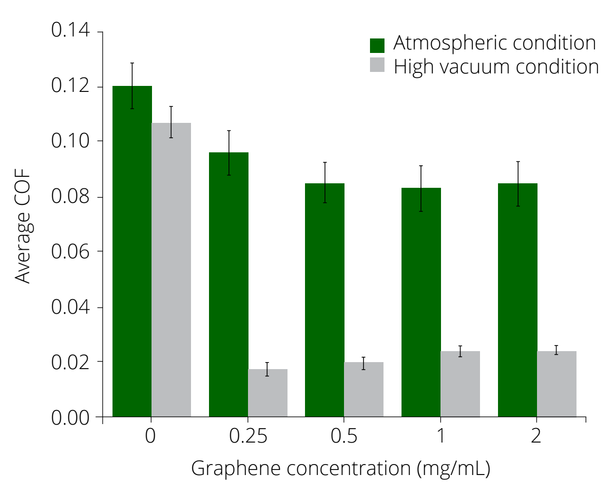

Figure 8: Average of coefficient of friction (COF) as a function of the concentration of graphene nanoparticles in the solution.

Source: Kolawole et al.[60].

For aeronautical applications, the main substrates under study were aluminum and its alloys. Due to their high adhesion in multilayers, it was possible to decrease the ice adhesion[65], and they resisted rigorous rain erosion tests[65]. While the conventional DLC film resists a maximum of 5 min in severe testing, DLC film obtained by pulsed DC PECVD with ion and electron confinement technique resisted over 60 min[65].

Figure 9 shows the results of ice adhesion tests on DLC-coated aluminum substrates, *i.e.*, the level of icephobicity, compared to the most used metals in aviation. Several samples with DLC deposited under various parameter sets were tested.

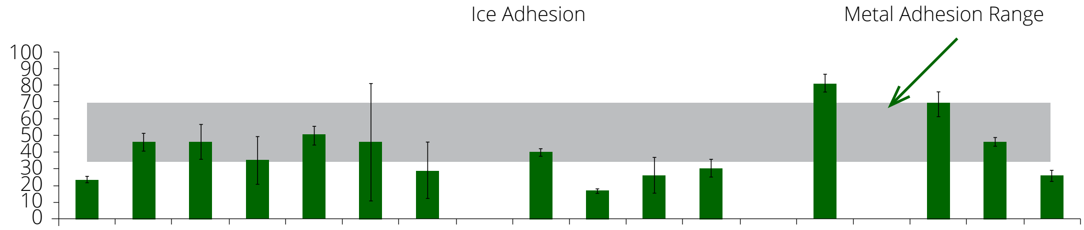

Figure 9: Ice adhesion tests on diamond-like carbon (DLC) surface in different samples. The vertical bars represent the measures of the adhesion coefficient of the samples with DLC, and the gray horizontal bar represents the range of the adhesion coefficient of the metals already tested.

Source: Bonetti and Trava-Airoldi[65].

It is observed that several DLC samples, including samples 2b and 4c, have adhesion coefficient below the gray range, showing better performance for this application. Figure 10 shows the results of rain erosion tests for these two specific samples. A test was applied in which a high density of water droplets is launched on the DLC surface at high speed (~800 m/s).

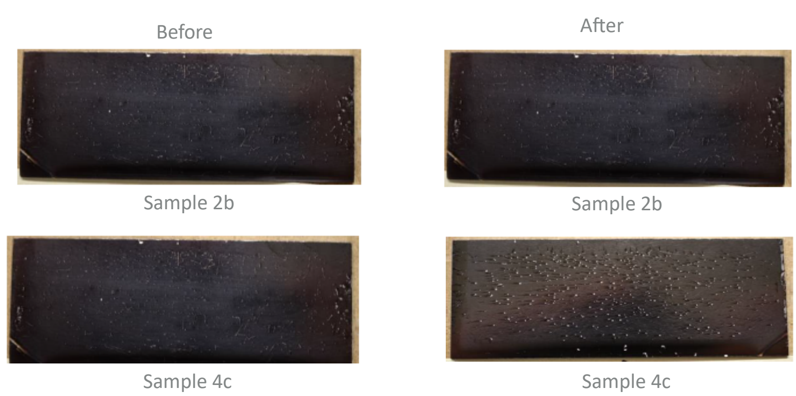

Figure 10: Samples 2b and 4c before and after rain erosion tests.

Source: Bonetti and Trava-Airoldi[65].

It is observed that for these two samples, after 60 min of continuous testing at maximum power, there was practically no destruction of the DLC film. Only small pits are observed, since the substrate is aluminum, which is a very soft material.

PECULIAR FEATURE OF THE PULSED DIRECT CURRENT PLASMA ENHANCED CHEMICAL VAPOR DEPOSITION TECHNIQUE WITH ADDITIONAL CATHODE

The main differential characteristic, in addition to improving all the properties of the film in relation to conventional techniques, is the growth temperature. Whereby controlling the growth parameters, it is possible to grow DLC film with good characteristics at a temperature as low as 50°C.

Besides aluminum, studies of adhesion of the DLC film on composite materials were carried out, with aeronautical applications and on polyethylene fiber, with medical and aeronautical applications, as shown in Fig. 11.

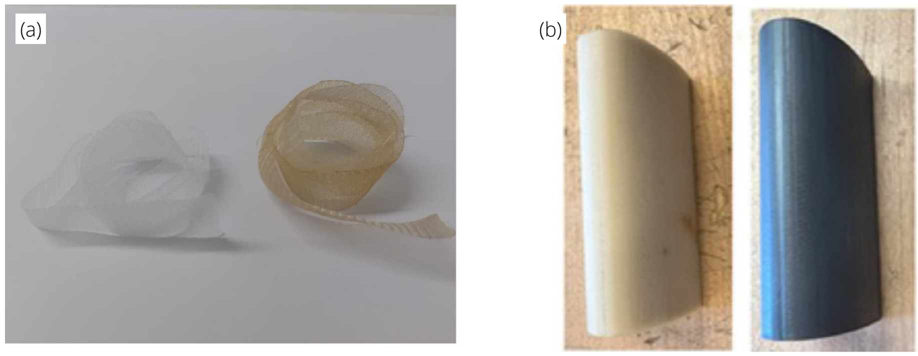

Figure 11: Adherent diamond-like carbon film on polyethylene and composite material. (a) Polyethylene fiber, before and after deposition of diamond-like carbon (DLC) with 200-nm thickness and (b) composite material, before and after deposition of DLC, with 5,000-nm thickness in five layers.

Source: Bonetti and Trava-Airoldi[45,65].

It can be seen from Fig. 11 that the deposition is uniform in any form of substrate, with thicknesses from a few nanometers to several micrometers.

Finally, the uniformity of DLC deposition in the three dimensions is symbolized in a large device with dimensions of about 500 mm and complex geometry, used in a protein processing system, as shown in Fig. 12[65].

Figure 12: Large device with complex geometry coated with diamond-like carbon.

Source: Bonetti and Trava-Airoldi[65].

Good uniformity in the deposition of the DLC film in multilayers shows the range of applications that can be considered with the pulsed DC PECVD technique with ion confinement system.

At this point, one can observe that the pulsed DC PECVD technique with ion and electron confinement system allows to improve all DLC properties depicting higher adhesion, lower coefficient of friction, higher hardness, higher thickness (multilayer film), etc., increasing a lot the fields of application.

BIOLOGICAL APPLICATION

Systematic research on DLC films as biomaterials began with the study by Anne-Thomson et al.[69], which established the biocompatibility of DLC and identified it as a biologically inert surface. *In-vitro* tests with peritoneal macrophages and mouse fibroblasts showed that DLC coatings did not induce adverse biochemical or morphological effects in cultured cells[69].

Reviews in the early 2000s summarized research on DLC films, with Hauert[70] highlighting that doping-modified DLC surfaces can exhibit tunable biological properties. The review indicated that changes in surface chemistry due to doping affect initial protein adsorption, which influences cellular response and hemocompatibility.

Roy and Lee[71] investigated DLC coatings for orthopedic applications, focusing on reducing wear and debris generation, and for cardiovascular applications to decrease thrombogenicity. They also reported inconsistent results, including cases in which DLC-coated stents did not show significant improvement, emphasizing the need to study the mechanisms responsible for both positive and negative outcomes.

In the cardiovascular field, Gutensohn et al.[72] demonstrated that DLC coatings function as passive diffusion barriers against electrochemical corrosion. The DLC layer on stainless steel stents serves as an impermeable chemical barrier, effectively preventing the release of nickel and chromium ions from the substrate, which are associated with thrombosis and inflammation. This resultes in minimal ion release and reduces thrombogenicity compared to uncoated stents.

Jones et al.[73] identified a complementary mechanism, attributing the hemocompatibility of DLC to its hydrophobic surface properties. The hydrophobicity facilitated selective protein adsorption, resulting in a higher ratio of adsorbed albumin to fibrinogen. This modulation of protein adsorption led to reduced platelet adhesion and activation on the DLC surface.

These findings established that DLC coatings in blood-contacting applications function through both passive ion barrier effects and active modulation of protein adsorption.

By the end of the decade, research moved from bioinertia to biofunctionalization, with the goal of designing active biological responses. Marciano et al.[74] introduced AgNPs addition to create Ag-DLC films with antibacterial properties. These films demonstrated efficacy against *Escherichia coli* and established DLC as a matrix for controlled therapeutic release.

Marciano et al.[75] investigated DLC with TiO_2NPs addition. TiO_2NPs, a photocatalytic bactericide activated by ultraviolet light, increased the hydrophilicity of the DLC surface and enhanced bacterial adhesion. The increased adhesion improved bactericidal efficacy by promoting greater contact between bacteria and the photocatalytic agent.

The demonstration of inert biocompatibility[69], elucidation of hemocompatibility mechanisms[72–74], and proof of concept for active functionalization[75,76] provide the foundation for the current research, which is addressed in the following review.

DOMAIN 1: ORTHOPEDIC AND DENTAL IMPLANTS (FOCUS ON BIOACTIVITY)

For orthopedic and dental applications, DLC coatings are primarily used to enhance bioactivity and support osseointegration. Incorporation of titanium dioxide nanoparticles modifies the surface physicochemical properties, resulting in increased surface free energy and hydrophilicity. These changes improve cell adhesion, as evidenced by higher viability (MTT assay), and enhance spreading of fibroblasts and osteoblasts[77–79]. Additionally, nanostructuring DLC films with nanodiamond (NCD) particles has proven effective, significantly increasing mitochondrial activity (viability) and reducing cell death, as indicated by decreased lactate dehydrogenase (LDH) release in L929 fibroblasts.

These findings suggest that nanodiamond incorporation enhances the biocompatibility of DLC films[80]. Furthermore, this strategy demonstrated strong *in-vivo* biocompatibility according to International Organization for Standardization (ISO) 10993-6, as it did not induce inflammatory responses in peritoneal implants[81].

For dental applications, DLC coatings with a silicon interlayer on Ti6AL4V abutments significantly improved mechanical and electrochemical properties. In artificial saliva, hardness increased from 1.83 to 11.81 GPa, and the coefficient of friction decreased from 0.20 to 0.04. The corrosion rate was reduced by a factor of 16, from 73.2×10^{-4} to 4.5×10^{-4} mpy[81].

DOMAIN 2: CARDIOVASCULAR APPLICATIONS (FOCUS ON BIO-INERTNESS)

In cardiovascular applications, the primary objective is to achieve complete bio-inertness to minimize platelet adhesion and reduce the risk of thrombosis. DLC coatings are increasingly investigated for being used in circulatory assist devices, in which direct blood contact requires careful control of the material-blood interface to prevent adverse effects such as thrombosis. As a result, advanced surface biofunctionalization is essential to address these challenges[82].

Sa et al.[82] investigated the relationship between surface properties and mechanical stability by evaluating textured titanium wires coated with TiO_2-DLC at varying thicknesses, implanted *in vivo* without anticoagulation. The thin DLC coating (0.3 μm), which was super-plasmophobic, exhibited delamination and wear, resulting in localized biological adhesion and neointima formation. In contrast, the thicker DLC coating (2.4 μm), which was plasmophilic, maintained mechanical stability and showed the lowest biological adhesion after 12 weeks.

This finding is significant, as it indicates that in high-flow applications, the structural integrity and thickness of the DLC coating may be more critical for long-term hemocompatibility than initial surface energy optimization. This result aligns with external studies on the Vroman effect. For example, Goyama et al.[83] reported that DLC coatings failed in 1.5-mm grafts due to increased fibrinogen adsorption ($p = 0.011$), which is pro-thrombotic and negated the intended benefits.

DOMAIN 3: ANTIMICROBIAL AND ANTI-BIOFILM SURFACES

The prevention of implant-associated infections is a key area in which functionalized DLC coatings are investigated. Early studies[76,84] demonstrated that pure DLC films (a-C:H) are not entirely inert but exhibit intrinsic bioactivity, resulting in significant cell membrane damage. This effect led to mortality rates of 28.4% for *E. coli* and 35.4% for *Staphylococcus aureus*[84].

The dominant research strategy has been to enhance this intrinsic bioactivity through elemental doping:

- Non-metals (F, TiO_2): Fluorine doping increased bactericidal activity to approximately 60%[85];
- Doping with TiO_2[75] increased surface hydrophilicity, which led to higher bacterial adhesion. Under ultraviolet irradiation, this increased adhesion resulted in improved overall bactericidal efficacy due to enhanced contact between bacteria and the surface;
- Metals and nanostructures (Ag, ND, ZrO_2, Zn): Ag-DLC coatings[74] showed increased antibacterial efficacy. Further studies with nanodiamond and zirconia coatings also demonstrated high antibacterial properties[86,87]. The Zn-DLC films achieved high antimicrobial efficacy, with approximately 100% reduction in *E. coli* and *S. aureus*, and 99.6% reduction in *Candida albicans*[88].

However, the study also demonstrated that Zn-DLC films were highly cytotoxic to oral keratinocytes when tested with 100% of the extract. On the other hand, diluted extracts (10%) from films with lower zinc content (43.4 at.%) showed no significant difference in cell viability compared to the uncoated control group, indicating that cytotoxicity can be mitigated by reducing the zinc concentration in the coating.

DOMAIN 4: BIOCOMPATIBILITY VALIDATION AND FAILURE ANALYSIS

In parallel with the development of specific applications, the DLC platform, particularly TiO_2-DLC and NCD-DLC composites, underwent validation through a rigorous biocompatibility pipeline in accordance with ISO 10993 standards:

- Phase 1: *In-vitro* screening (ISO 10993-5): Initial investigations[80] assessed the cytotoxicity of the nanocomposites. Employing the standard L-929 fibroblast model, these studies demonstrated that TiO_2 and NCD nanocomposites were biocompatible. Specifically, NCD-DLC increased cell viability (MTT assay) from approximately 58 to 85% and reduced cell death (LDH assay) from 68 to 44% relative to pure DLC[80];
- Phase 2: *In-vivo* screening (ISO 10993-6): Wachesk et al.89 advanced the safety assessment by conducting *in-vivo* tests at a non-application-specific site. The implantation of TiO_2-DLC films in the peritoneal cavity of

mice, followed by evaluation of the inflammatory response via macrophage lavage, confirmed that the films "did not induce an inflammatory process;"

- Phase 3: Failure modes (corrosion): The research further examined potential failure modes[90–93]. Ramos et al.[90] investigated electrochemical corrosion resistance using simulated body fluid as an aggressive electrolyte. Analysis through electrochemical impedance spectroscopy and Nyquist/Bode plots confirmed the stability of NCD-DLC films, which is essential for implantable devices.

This systematic validation pipeline, which progresses from general *in-vitro* to *in-vivo* and application-specific testing, exemplifies a robust methodology in biomaterials engineering.

NEW BIOLOGICAL APPLICATION: DIAMOND-LIKE CARBON FOR SURFACE-ENHANCED RAMAN SPECTROSCOPY

Molecular-level detection has become an important part of medical diagnostics, disease and environmental monitoring, forensic science, and food safety testing. This technique ensures a rapid and sensitive method to detect traces in very small volumes and in complex environments[94]. The identification of target analytes allows us to obtain information about cellular functions and evaluate therapeutic efficacy.

Most times, immunological or molecular biology methods, such as enzyme-linked immunosorbent assay or polymerase chain reaction, are applied to detect peptides and proteins, as well as to amplify nucleic acids for reliable and sequence-specific detection, respectively[95,96]. Optical methods have been extensively studied[97,98] for bioanalytical purposes even though some techniques require high operating expenses. Magnetic and plasmonic analysis techniques are some of the other methods implemented in laboratories for rapid and sensitive detection of trace analytes.

Over the past 10 years, the ultrasensitive detection platform based on SERS has received considerable attention as a promising approach for target molecule detection due to its high specificity, improved sensitivity, and rapid readout capability[94,99–102]. SERS is a vibrational optical technique that boosts the scattered light signal by several orders of magnitude compared to the regular Raman signal and can identify analyte molecules at very low concentrations, even single-molecule detection[103]. The high sensitivity of SERS is due to the localized surface plasmon resonances produced by noble metal nanostructures, particularly silver and gold[104]. The efficiency of a SERS substrate is largely dependent on surface characteristics. As a result, the fabrication of robust, repeatable, and high-sensitivity SERS substrates in which the plasmonic resonance effect can be optimized remains a challenging research topic[105].

The SERS technique has many applications, including biological sensing, trace analysis, medical diagnostics, forensic science, and the detection of pesticides, explosives, and drugs, among others[106,107]. Recently, SERS has been applied primarily in bioanalytics, in which it has been used to study nucleotides and amino acids, including complex functional structures, such as nucleic acids and proteins[102,108,109]. In addition, SERS is an attractive tool for investigating and characterizing whole prokaryotic cells, such as bacteria, biofilms, viruses, eukaryotic cells, and tissues[110].

Despite significant advances in SERS substrates for biomolecule detection, manufacturing solid substrates with highly sensitive, efficient nanostructured surfaces remains a major challenge due, among other factors, to the analyte-nanostructure-surface interaction that generates "hot spot" regions in which the Raman signal are amplified. For some types of molecules, it is possible to detect concentrations as low as 10^{-12} mol/L[111–113].

Given the need to fabricate solid SERS substrates, in which the relationship among the substrate's platform, metallic nanostructures, and analyte interaction enables plasmonic resonance to enhance Raman sensitivity, a DLC-based substrate covered by AgNPs was recently developed[68]. Figures 13a and 13b show field emission gun-scanning electron microscopy (FEG-SEM) images of the nanostructure of the DLC nanoparticles at different magnifications, in which we can see the rough structure obtained after laser treatment of the DLC film.

The DLC film, laser-treated, forms a rough structure with DLC nanoparticles ~100 nm in size. After silver electrodeposition on DLC, FEG-SEM micrographs show that AgNPs were deposited throughout the DLC structure (~85 ± 0.3% are silver covered), as shown in Fig. 13c. AgNPs form agglomerates of various sizes and do not have a defined spherical structure (Fig. 13d).

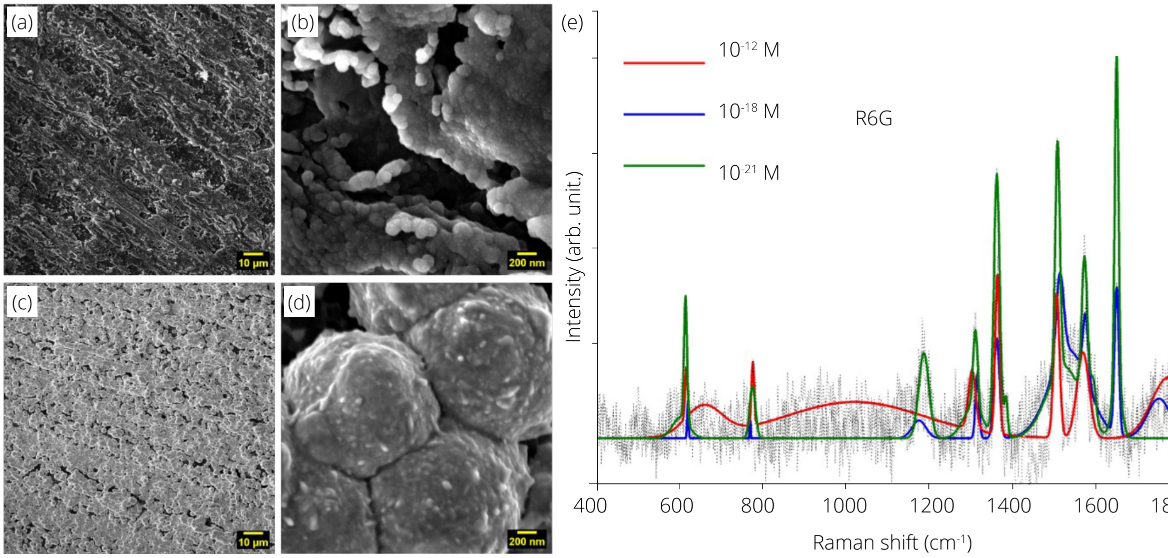

Figure 13: R6G on surface-enhanced Raman spectroscopy (SERS) substrate. (a, b) Field emission gun-scanning electron microscopy (FEG-SEM) images for diamond-like carbon (DLC), and (c, d) for DLC/Ag. SERS spectra of the R6G molecule at different molar concentrations, and (e) the SERS spectra for three different concentrations of R6G molecules in water solution.

Source: Washek et al.[68].

To evaluate the sensitivity of the DLC/Ag SERS substrate, Raman spectroscopy was employed to detect R6G at concentrations of 10^{-12}, 10^{-18}, and 10^{-21} M. The SERS spectra is presented in Fig. 13e, in which the principal peaks of R6G are displayed with high intensity. At the concentration of 10^{-12} M, there is a notable increase in analyte fluorescence. A significant finding is the successful detection of R6G at 10-21 M, as illustrated in Fig. 13e, which confirms the promising efficiency and single-molecule detection of the DLC/Ag SERS substrate.

Given these unexpected results, the DLC/Ag substrate is currently tested to determine the limit of detection for various substances, with the potential for applications in medical and biological fields, agriculture, and environmental monitoring, among others.

APPLICATIONS OF DIAMOND-LIKE CARBON INSIDE OF TUBES

The protection of internal metallic surfaces in tubular components using DLC coatings has gained increasing relevance due to the need to mitigate degradation under corrosive, erosive, or abrasive environments. Such technique can be applied in a wide range of engineering components, including engine cylinders, aircraft landing gear assemblies, military gun barrels, pipelines for petroleum and chemical transport, among others[114]. Wear occurring on internal surfaces can lead to significant reductions in operational performance and, in severe cases, catastrophic failure, with associated economic consequences. For these reasons, protective coatings for internal geometries remain a subject of considerable technological interest, although their practical implementation is still challenging due to the geometric constraints inherent to tubular substrates.

Diamond-like carbon inside of small tube

The physics of electrical discharges within tubular interiors has been extensively studied since the 1930s[115], with the hollow cathode discharge (HCD) emerging as the predominant technique. Despite its elevated plasma density, the HCD method is generally not ideal for the growth of high-quality DLC films. This has motivated the development of alternative methods capable of maintaining non-thermal plasma equilibrium. Notable examples include PECVD[116], plasma immersion ion implantation (PIII)[117], magnetic field enhanced plasma deposition[118], and PIII&D in crossed fields[119]. Several innovations have been introduced for DLC deposition, including the incorporation of microwave antennas in hybrid configurations[120]; the strategic use of auxiliary electrodes to mitigate potential drops and enhance plasma uniformity[120]; and the application of magnetic fields[119].

Deposition uniformity remains an important challenge reported in numerous studies of tubular substrates, primarily due to the reduction of ion energy[121,122]. This effect arises from the complex behavior of the plasma sheath and is directly correlated with the relationship between the sheath overlap radius (D) and the substrate diameter (R)[123]. This functional dependence is regarded as a characteristic parameter of discharges within tubes, given by Eq. 1:

$$D = \sqrt{\frac{-4\varepsilon_0\phi_0}{en_0}} \quad (1)$$

where: ε_0: the permittivity of free space; φ_0: the applied voltage (potential) at the tube; *e*: the elementary electric charge; n_0: the bulk plasma density.

The existence of plasma inside the tube is guaranteed when the condition R > D is satisfied.

Further challenges arise during DLC deposition inside tubes that compromise both coating quality and uniformity.

The first major difficulty is the edge effect, which leads to plasma intensification at the tube extremities, causing excessive ion bombardment and localized overheating. This phenomenon adversely affects the homogeneity of the deposited DLC relative to the central region and simultaneously promotes sputtering, resulting in cross-contamination of the growing film by metallic species released from the tube surface itself[124].

A second critical challenge arises from the need to maintain a uniform plasma density along the axial length of tubes with a high aspect ratio ($L \gg D$), which significantly limits process scalability[125]. Overcoming these limitations requires intricate optimization of the power supply and precursor gas configuration, together with incorporating additional gas injection points to ensure a homogeneous process feed. These persistent challenges underscore the continuing need for research in surface engineering aimed at achieving reliable and uniform DLC deposition.

Diamond-like carbon inside of long tubes

Plasma-based coating processes applied to the interior surfaces of tubular components are typically carried out within a vacuum chamber. This requirement inherently limits the practical treatment of long tubes (often several meters in length) as it necessitates a vacuum system larger than the tube itself. From an economic standpoint, scaling up to chambers of such dimensions is frequently prohibitive. Consequently, this technological constraint has motivated the development of alternative strategies, most notably the concept of using the tube itself as the deposition chamber for DLC film growth, as proposed by Trava-Airoldi et al.[126] and Casserly et al.[127] and shown in Fig. 14.

Crucial factors governing the successful deposition of coatings onto the inner surfaces of tubes have been well documented in the literature. It has been established that employing a unidirectional gas flow (gas feeding and evacuation occuring in the same direction) often leads to complex plasma behavior and the formation of non-uniform coatings[127]. This issue can be effectively mitigated by implementing a bidirectional gas flow configuration, in which the gas is supplied and evacuated at both ends of the tube, resulting in thicker DLC films with improved quality[128,129]. Furthermore, plasma operating parameters play a decisive role, as demonstrated by Wang et al.[130], who showed that the structural properties of DLC films are strongly influenced by the internal gas pressure. However, high-pressure operation presents a critical challenge, as the plasma discharge tends to evolve into an arc discharge. This instability was successfully addressed by Iwamoto et al.[131], who achieved stable plasma generation glow through the application of nanopulses to the high-pressure plasma discharge.

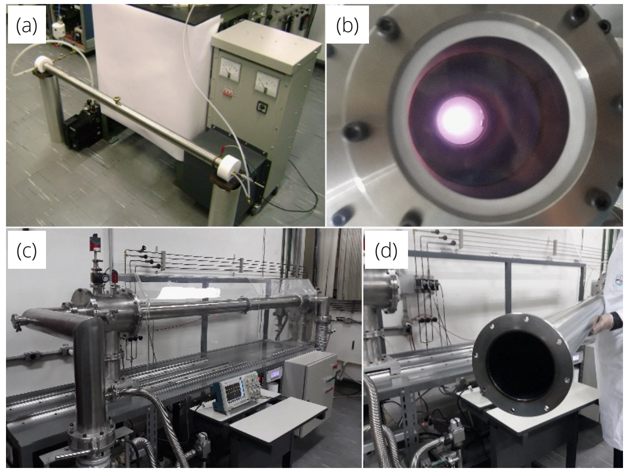

Figure 14: Pulsed direct current plasma enhanced chemical vapor deposition for diamond-like carbon (DLC) films deposition at the National Institute for Space Research, Brazil: (a) preliminary experimental setup with a 1,300-mm long and 50-mm diameter tube; (b) typical plasma discharge observed inside the tube; (c) enhanced operational system, consisting of a tube 2,000 mm in length and 100 mm in diameter; (d) tube after DLC coating.

Source: Pillaca et al.[128].

Therefore, it has been demonstrated that it is possible to obtain a DLC film of good quality and good uniformity inside long tubes. Further study is needed regarding the dependence of its properties on the tube/reactor aspect ratio, which is defined as the ratio of the tube diameter to its length.

CONCLUSIONS AND FUTURE PERSPECTIVES

This work highlights the broad adoption and significant performance enhancements of DLC coatings by using a pulsed DC PECVD technique, more specifically through the pulsed DC PECVD technique with ion and electron confinement system. This improved technique expands DLC applications into challenging environments and specialized fields, particularly aerospace and biomaterials engineering. As mentioned, the key performance advancements concerning the addition of the ion and electron confinement system dramatically improves the mechanical and tribological properties of the DLC films, allowing for:

- Higher adhesion on a wide range of materials, including soft metals (like aluminum), non-metallic materials (composites, carbon fibers), and polymers (due to the unique ability to grow films at low temperatures);
- Enhanced performance, like thicker, more flexible films, and a lower coefficient of friction mainly in ultra-high-vacuum environments (critical for space applications).

Concerning biomedical application, DLC is highly valued in biomaterials engineering due to its tunable properties. Its performance is dictated by its hybridization structure, chemical composition, and the specific application environment, necessitating tailored formulations rather than a universal coating.

On implants (orthopedic and dental), the DLC enhances mechanical stability and all kinds of bioactivity. Also, applied on cardiovascular devices, it achieves bio-inertness and minimizes adverse reactions, which are strongly influenced by protein adsorption (the Vroman effect).

Concerning antimicrobial surfaces application, the DLC film from pulsed DC PECVD with ion and electron confinement system shows potential by inhibiting biofilm formation, as the film can be deposited without any kind of metallic dopants, and so, without cytotoxic effects.

Moreover, the coatings exhibit aeronautic properties like rain erosion resistance and icephobic properties. Also, the deposition process can be applied inside long tubes for aggressive liquid transport systems.

For the future, new rote needs to be studied, concerning biologic research on advanced coatings with functionalized surface design to meet the specific requirements of complex biomedical devices, such as neural interfaces and incorporated nanotechnology. The pulsed DC PECVD technique with ion and electron confinement offers the possibility of very big scaling up allowing deposition on big and complex threedimensiona surfaces.

CONFLICT OF INTEREST

Nothing to declare.

AUTHOR CONTRIBUTIONS

Conceptualization, Project administration, Writing – review & editing: Trava Airoldi VJ, Bonetti LF; **Writing – original draft, Formal Analysis, Resources, Methodology:** Bonetti LF, Marciano FR, Lima GG, Monção RM, Correa LSM, Pillaca EJDM, Corat EJ, Trava Airoldi VJ. **Supervision:** Trava-Airoldi VJ; **Final approval:** Trava-Airoldi VJ.

DECLARATION OF USE OF INTELLIGENCE ARTIFICIAL TOOLS

The writing of this review paper did not use artificial intelligence.

AVAILABILITY OF DATA AND MATERIALS

The data will be available upon request.

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