

# Cheap Diamond-Like Carbon (DLC) Thin Films: Nanotechnology Transfer from Academia to Industry

Professor Elvira Williams  
Founder, Owner, and Lead Scientist, Syntraguard, LLC  
Greensboro, North Carolina, USA  
El.Williams@Syntraguard.com, welviraw1@yahoo.com

**Abstract--This document is a report by Syntraguard, LLC, a high-tech Center for Interdisciplinary Research and Development, that is meant to illustrate a practical means of nanotechnology transfer from academia to industry through partnering. The core competency of Syntraguard is deposition of thin films of DLC (diamond-like carbon) on various client-provided substrates tailored to specific client needs. Because of their superlative characteristics in almost every area for which materials are considered, DLC films offer the best hope for the fabrication of various types of advanced materials and devices that meet the nation's and world's projected technology goals for the 21st Century. Continuous development and use of other competencies outside its core are needed to fulfil the broad mission of Syntraguard.**

**Keywords -- DLC, diamond-like carbon, synthetic diamond, diamond thin films**

## I. INTRODUCTION

Syntraguard, LLC is now solely owned and operated by professor Elvira Williams. It began as a high-tech start-up Center for Interdisciplinary Research and Development MWBE (Minority Women Business Enterprise) owned and operated by three women with long-term careers in the academic and business worlds. It is a scientifically-based solution provider. Its mission is to Creatively Research, Develop, Engineer, Re-engineer, and/or Refine Scientifically Based Technologies Resulting in Processes, Products and Usages for the Current and Future Benefit of All Humanity and the Global Environment. The core competency and primary focus of Syntraguard is low-cost successful deposition of thin films of DLC (diamond-like carbon) on various client-provided materials as substrates. Syntraguard was created around its core competency skill. This skill is among those brought to Syntraguard by the current owner/operator who has a long-term background in academic research - essentially demonstrating a transfer of nanotechnology research potential from academia to industry,

through partnering, to help Syntraguard fulfil its total mission. Major focus clients include the military, airlines and automotive firms facing costly and potentially hazardous erosion of moving parts, as well as, other corporations producing moving parts made and sold to still other corporations and to the public. Typical surfaces coated include tools for cutting, moulding, and forming still other tools. They also include automotive, industrial, aerospace, and medical components. The need for continuing innovation creates a futuristic timeline for these services that is heavily shaped and destined only by the imagination.

Diamond-like carbon (DLC) films are metastable amorphous materials, containing a mixture of  $sp^2$ ,  $sp^3$  and sometimes  $sp^1$  coordinated carbon atoms in a disordered network.

DLC films are characterized by a spectrum of interesting properties which include extreme hardness, a generally low friction coefficient, high optical transparency over a wide spectral range, high electrical resistivity, and chemical inertness to both acids and alkalis. The term diamond-like is used for these materials because, on average, their properties are similar to those of diamond. The attractive properties of diamond-like carbon have stimulated wide interest in characterization and practical applications of DLC, such as wear-resistance coatings, corrosion protection of metals, optical equipment, lubricity, resistance to galvanic activity, electronic components, or as a dielectric material [1].

DLC films exhibit superlative characteristics in almost every area for which materials are sought. Therefore, DLC films offer the best hope for the fabrication of various types of advanced materials and devices that meet the nation's and the world's projected technology goals for the 21st Century.

## II. EXPERIMENTAL TECHNIQUES

A schematic and a photograph of the RF-PECVD apparatus used to fabricate the DLC described in this report are shown in Fig1. In this process, a reactant gas mixture is allowed to flow into an evacuated deposition chamber which is designed as a cylindrical parallel plate capacitor, as illustrated in the schematic in Fig. 1. The electric field

(arrows) produced between the plates by a radio frequency (RF) power supply connected to the chamber, provides the energy to create a plasma between the plates. Solid DLC films deposit on substrates (placed on bottom plate) as the plasma flows over them as it is being drawn into the exhaust port in the center of the bottom plate. The open-lid photograph view of the deposition system in Fig. 1 shows the exhaust port in the center of the bottom plate (equipped with temperature control) surrounded by wafers as substrates on which DLC is to be deposited. The chamber was manufactured by Technics, Inc., now PETS (Plasma Equipment Technical Services Inc.). It has a pyrex window (viewport) on the lid for observing (through the closed lid) the chemical reactions (with characteristic purplish glow) that produce the DLC films.

The basic system is a 16" Diameter Anodized Aluminum Chamber with, 4 gas channels and a low Frequency (40 KHz) Power Supply delivered to a water-cooled electrode. Its Footprint is 17 1/4" X 19 1/2" X 20" High. The system uses a Mechanical Pump (Rotary Vane filled with Fomblin Oil). It has connecting hose (KF-40 stainless steel convoluted bellows) and fittings to allow for vacuum connection of Pumping Package to the main system vacuum bulkhead fitting. It has 4 channels of Mass Flow (0 to 100 SCCM) including gas manifolds and Nupro pneumatic isolation valves. It has an Advanced Energy PE-1000, 1250 watt, 40 KHz power supply (air cooled) for driven electrode RF excitation. It has upside down shower head functionalized for uniform gas mixture over the heated platen (+ or - 5% Uniformity guarantee). The 16-inch Anodized Aluminum Chamber with water-cooled top and base plate cooling circuit has capability of being heated as long as a heater/chiller is utilized, 22 degrees to 80 degrees centigrade. The O. D. (outer diameter) of the Stainless Steel Lower Heated Platen is 12 inches. A 1500 watt cal rod heater is contained within the lower heated platen assembly. The assembly has a Eurotherm PID Controller for controlling temperature of the heated

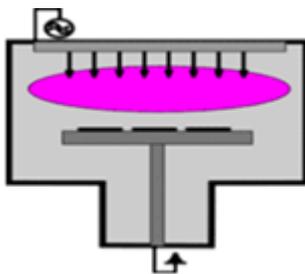
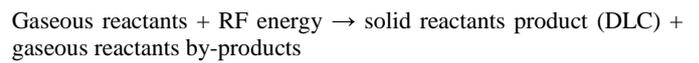


Fig. 1. RF plasma-enhanced CVD chamber/reactor platen. The system is equipped with a MKS Model 122A Baratron Gage for vacuum measurement (10 Torr head). It has VCR fittings for Process Gases and Vent Gas Line. Water IN & Out and compressed Air lines use 1/4 - inch Swagelok Fittings. A 208 VAC, 3 Phase, 60 Hz, 30 Amp service is required for the main console (all power requirements are distributed with the unit. Definition for 3 phase power is two hots, one neutral & one ground wire).

Reactant gases in proprietary formulation have been successfully used, over the years, with Syntraguard apparatus to produce quality DLC films on various substrates; with results presented at various national and international conferences, seminars, and workshops [2] – [5]. Under appropriate controlled conditions of temperature, pressure, composition, and RF power, the solid DLC films form on the substrates while gaseous by-products are released through the exhaust, according to the General Reaction:



Syntraguard will scientifically create new warranty avenues utilizing CVD chambers it uniquely designs. Scientific and engineering diversification is planned to meet current world challenges, such as producing clean water in third world countries. DLC must be distinguished from diamond layers, which are made by different CVD methods. The diamond films are polycrystalline materials with crystallites up to tens of micrometers in size, having diamond crystal structure. Under bombardment with high-energy ions, DLC films may crystallize [6] and some degree of crystallinity has been reported for films deposited at relatively high substrate bias [7], although DLC films are essentially amorphous materials without any long-range order. However, short- or medium-range order may occur in these films [8]. DLC thin films cover a wide range of characteristics between those of diamond, graphite, and hydrocarbon polymers. The term diamond-like is used for these materials mainly because, on average, their properties are most similar to those of diamond.

To understand the physical basis of the properties of DLC, in particular those fundamental attributes that determine resistance to physical and chemical attack, one must characterize the basic atomic structure of the material. The ratio of carbon atoms in each bonding coordination, the hydrogen content of the film, and its distribution in the bulk of the film are all very important. The characterization of the physical properties of DLC films does not always predict their frictional behavior or thermal stability. These two can be affected by deposition conditions in a way not reflected by physical characterization, especially wear characterization, required when films are to be used for wear protection purposes.

Although it is difficult to achieve, hydrogen-free amorphous carbon films can be obtained [9]. However, the incorporation of hydrogen properties, into the character of the film is usually a must. As a function of deposition method,

the hydrogen content in DLC films has been found to vary from less than 10% [10] up to 60% at the atomic level and, therefore, the physical properties of the films also vary [11]. Hydrogen content is also key to obtaining a wide optical gap and high electrical resistivity, because it passivates the dangling bonds in the amorphous structure [12]. The  $sp^3$  (sigma) carbon bond is characterized by a lower binding energy than  $sp^2$  (pi) bonds, therefore dangling electronic orbitals of amorphous carbon and unpaired electrons, create states in the energy gap between bonding and antibonding. Hydrogen removes these states from the gap by closing dangling bonds and reduces the density of graphitic states [7], where it was found that the  $sp^3 : sp^2$  ratio increases with increasing hydrogen concentration. It was also found that DLC films containing less than 50% hydrogen were harder than those containing more hydrogen. According to Angus and Jansen (1988) [12], to obtain diamond-like properties in films deposited from hydrocarbons, the films have to include hydrogen in concentrations from 17%-61%. There has been a suggestion that significant quantities of free hydrogen may exist in DLC layers, either bound in clusters [13], or intercalated into turbostratic graphite layers [14] embedded in an amorphous matrix.

### III. DISCUSSION

Although the core competency and primary focus of Syntraguard is successful deposition of thin films of DLC (diamond-like carbon) on various client-provided substrates, another important goal of Syntraguard is to create job opportunities within its scope of expertise for disenfranchised, disadvantaged, and marginalized portions of the population. Syntraguard's business philosophy is to form and grow companies of scientists, engineers, and business professionals to create new products, processes, and services for the benefit of all humankind. As part of this effort, Syntraguard facilitates the growth and fosters the development of new technologies through the establishment of its STEM (Science Technology, Engineering, and Mathematics) apprenticeship and internship laboratory serving undergraduate and graduate students, as well as other interested persons. Such individuals are interviewed and invited to participate in lab environments and processes under supervision of scientists and engineers at Syntraguard. This gives participants an opportunity to work in a real world environment enabling them to be better prepared to compete in the global economy.

One very interesting, recently-added area of Syntraguard endeavor towards fulfilment of its overall mission, other than DLC production, is that of charcoal production from hardwood trees. This component was added during Summer 2016. So far, oak, walnut and pecan have been successfully used to produce charcoal. Charcoal is useful in water purification, a cited global interest of Syntraguard.

The owner/operator of Syntraguard has maintained certain pertinent professional partnering connections that were in place prior to establishment of the company for the purpose of enhancing its overall mission. These connections

include academic and other collaborations implemented through the STEM laboratory component of Syntraguard. These collaborations include Gateway University Research Park, formerly JNNRC (Joint Nanoscience and Nanotechnology Research Center), and NCA&T (North Carolina Agricultural and Technical State University), both in Greensboro, North Carolina, USA; NC (North Carolina) State University and Shaw University, both in Raleigh, North Carolina, USA; and NASA (National Aeronautics and Space Administration), headquarters in Washington, DC, USA.

Gateway University Research Park: The Gateway University Research Park provides world-class laboratories and office space to businesses, universities and the local community. In its uniquely collaborative environment, the tenants and partners utilize shared resources for technological growth, discovery and progress. Gateway's premier campus also provides the support needed to help turn cutting-edge intellectual property into thriving businesses in areas of life and physical science, engineering and other applied sciences. NCA&T: NCA&T is a public, coeducational, historically black, research university well-known for its outstanding engineering and other special technical programs. NC State University: NC State is a public, coeducational, research university well-known for its outstanding engineering and other technical programs. Shaw University: Shaw University is a private, 4-yr. coeducational, historically black, university. It is well-known as the first historically black, university in the southern part of the United States. It offers bachelor degrees in STEM and up through the masters degree in other areas. NASA: NASA is the United States government agency responsible for the civilian space program as well as aeronautics and aerospace research.

Before the Syntraguard operation was transferred from academia, it was used at both NCA&T and Shaw Universities to establish special programs and projects for STEM faculty and students at these schools. The PENS (Partnership for Excellence in the Natural Sciences) was established at NCA&T. Syntraguard incorporates various components of this program into its STEM laboratory.

The PENS Program is a model academic support retention and graduate studies preparation program with basic sponsorship through contracts, grants, and philanthropy. During one ten-year period, it was noted that PENS was especially successful (99.2 % retention of participants in a STEM major up through the completion of the undergraduate degree; 100% of which entered a STEM career with more than 50% of these earning Masters and PhD degrees). The overall PENS program goals: #1. To improve retention and #2. To increase the number/percentage of minority undergraduate students who: #1. successfully completed the requirements for a baccalaureate degree in STEM and related fields, and #2. who had the academic preparation, career awareness and motivation to successfully pursue graduate studies at the masters and Ph.D. levels. The specific objectives of PENS: #1. To recruit highly qualified students into the PENS Interns Program, #2. To develop and implement academic support services for students that

strengthened and/or developed the academic (and character) skills that would provide enrichment for the undergraduate experience, #3. To develop and implement services that promoted student-faculty interaction through research and mentoring, and #4. To develop and implement linkages for PENS Interns with existing NCA&T University research and other academic enrichment programs, doctoral-granting universities, national industrial and government laboratories, and other sources. Workshops and Seminars (e. g., Study Skills, Graduate Record Examination Preparation). It has been noted that during one ten-year period, more than 125 students were successfully put into the pipeline towards masters and PhD degrees through this program, and all have entered successful STEM careers.

A Nanoscience and Nanotechnology Research (NNRC) Initiative was established at Shaw University. The mission of the Shaw NNRC was: To promote and enhance academic excellence at Shaw University, in research and teaching, through a cutting-edge integrated science approach. It had 4 divisions (each division headed by a world-class scientist): Nanobiology and Environment Research; Nano-Optoelectronic Materials and Devices Research; Computational Materials and Atomistic Engineering Research; and Education and Outreach. The NNRC sought to significantly elevate the educational prospects of Shaw University science students. Through the NNRC, STEM majors would go from earning standard B.S. degrees in the STEM areas of Biology, Chemistry, Computer Science, Environmental Science, Mathematics, and Physics to additionally earning B. S., M.S. and Ph.D. degrees with extensive research experience and expertise in the emerging fields of nanoscale science and technology. The NNRC sought to promote Shaw University's emphasis on holistic character education to prepare its graduates for life. The goals of the NNRC were: #1. Bringing Shaw University to the forefront of scientific endeavors in the nanoscience and nanotechnology areas, #2. Enhancing students' creativity, academic achievements and access to highly competitive opportunities through research and research training, #3. Upgrading the academic level of Shaw University STEM students through the introduction of Nanoscience/Nanotechnology related courses, #4. Upgrading the educational prospectus of Shaw University STEM majors beyond the Bachelor degree level and #5. Increasing the University capacity in terms of faculty and post-docs, and community outreach.

NASA: NASA provided a grant to Shaw in support of establishing the Shaw NNRC. The proposed objectives of the project were: #1. To establish an interdisciplinary nanoscience and nanotechnology research facility at Shaw University through partnering, and #2. To use the facility to train a group of 24 STEM (Science, Technology, Engineering, and Mathematics) faculty and students in aspects of nanoscience and nanotechnology research over the period of the grant. The project was successful. The NNRC was established in fulfillment of objective #1, and 34 individuals were trained in the facility in fulfillment of objective number

#2, which exceeded the projected number by 41.25%. The nanoscience and nanotechnology research techniques shared with STEM faculty and students at Shaw to set up and operate the NNRC were those acquired by the principal investigator of the NASA grant (now Lead Scientist at Syntraguard and lifetime NASA ambassador) while a NAFP (NASA Administrator's Fellowship Program) Fellow at NASA-Langley Research Center in Hampton, Virginia, USA.

#### IV. CONCLUSION

Syntraguard functions successfully at the research junction between academia and industry through the cited collaborations. With still other new collaborations (to be formed), Syntraguard seeks to fulfill its broad mission: to Creatively Research, Develop, Engineer, Re-engineer, and/or Refine Scientifically Based Technologies Resulting in Processes, Products and Usages for the Current and Future Benefit of All Humanity and the Global Environment. It aims to help develop the large projected high-tech workforce (through its STEM laboratory component) needed to keep pace with escalating technology-driven advancements of the 21st Century, as it simultaneously executes its function as a high-tech DLC coating enterprise. Its recently-added charcoal production component to the STEM laboratory is evidence of the determination of Syntraguard to have its activities keep pace with its purpose.

#### V. ACKNOWLEDGMENTS

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