

# Electrical and optical properties of diamond-like carbon

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## Abstract

Diamond-like carbon (DLC) films, amorphous hydrogenated or nonhydrogenated forms of carbon, are metastable amorphous materials characterized by a range of attractive mechanical, chemical, tribological, as well as optical and electrical properties. The films can be prepared at low temperatures, from a large variety of precursors, by a diversity of techniques, and their characteristics can be modified by incorporation of different elements such as N, F, Si, or metals. The diversity of methods used for the deposition of DLC films provides the flexibility to tailor their properties according to specific needs and potential applications. The optical gap of these materials is in the range of 1–4 eV and the electrical resistivity spans 14 orders of magnitude. The dielectric constant of DLC films covers the range of 2.5–6. The talk will review the optical and electrical characteristics of DLC and discuss the actual and potential applications based on these properties. © 1999 Elsevier Science S.A. All rights reserved.

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## 1. Introduction

Diamond-like carbon is a name attributed to a variety of amorphous carbon materials containing from less than 1 at% to about 50 at% hydrogen. The diamond-like carbon films contain significant fractions of  $sp^3$  type C bonds, giving them attractive physical and mechanical properties, some similar to a certain extent to diamond. Most of the hydrogenated films contain  $sp^3$  fractions usually smaller than 50%, while the nonhydrogenated carbon films can contain up to 85%  $sp^3$  bonds [1]. The correct terminology to be used to define the two types of films is still being disputed; however, the present paper will adopt the DLC term to designate the hydrogenated form of diamond-like carbon and the tetrahedral carbon (ta-C) term to designate the nonhydrogenated diamond-like carbon containing high fractions of  $sp^3$  hybridized carbon.

Both DLC and ta-C are metastable materials which have to be prepared under ion bombardment of the growing films. General reviews of diamond-like carbon materials can be found elsewhere [1,2]. The present paper will concentrate on the electrical and optical properties of diamond-like carbon materials and their applications in these areas.

## 2. Electrical properties and applications

### 2.1. Electrical properties

The electrical properties of diamond-like carbon films can vary from that of a semimetal to that of a wide bandgap insulator. Despite having a modest bandgap (1–4 eV), DLC or ta-C films do not behave like typical semiconductors. The electrical properties of diamond-like carbon have been modeled assuming a band structure consisting of only a mobility gap, where carriers residing in gap states are localized [3,4]. This mobility gap produces semiconductor behavior, however, the high density of localized gap states leads to low apparent carrier mobilities and significantly degrades the semiconducting properties of materials. The mobility at room temperature of both DLC and ta-C was found in the range of  $10^{-11}$ – $10^{-12}$   $\text{cm}^2/\text{V}$  [5]. The films are generally characterized by high electrical resistivities spanning a large range of values, from  $10^2$  to  $10^{16}$   $\Omega$  cm, depending on the deposition conditions [2]. The electrical resistivity of a specific type of diamond-like carbon can be strongly reduced by several orders of magnitude through incorporation of metals or nitrogen in the films [6,7]. However, the doping efficiency is very low and there is no evidence that it is possible to form pn junctions with diamond-like carbon films. It has been claimed that the decrease of resistivity by incorporation of dopants may be related to a dopant induced graphitization [8].

Doping was better identified in ta-C films where nitrogen

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was found to be a good *n*-type dopant causing a shift of the Fermi level from 0.91 eV above the valence band to 0.65 eV below the conduction band with increasing nitrogen concentration [9]. The density of states near the Fermi level extracted from the hopping transport parameters was found in the range of  $6.5 \times 10^{17}$ – $9.7 \times 10^{19}$  cm<sup>-3</sup> eV<sup>-1</sup>. The doping was explained by substitution in the *sp*<sup>3</sup> coordination at low N concentrations and adoption of *sp*<sup>2</sup> bonding at high N concentrations [9]. However, even in ta-C the doping efficiency is low due to ta-C's high intrinsic defect density. The low doping efficiency of taN has been explained by theoretical calculations of the total energies of various dopant and dopant/bonding combinations and it was shown that charged dangling bonds are less favored because of the stronger Coulombic repulsion in ta-C [8]. The dopants can instead be compensated by weak bond states in the lower gap associated with odd-membered  $\pi$ -rings or odd-numbered  $\pi$ -chains.

The electronic transport mechanism was investigated in pulsed plasma deposited (PLD) ta-C by correlating the effect of annealing on electrical conductivity and stress relaxation and using the latter to estimate the changes in *sp*<sup>2</sup> carbon concentrations [10]. The results indicated that, in the specific films, the conductivity is an exponential function of the changes in the *sp*<sup>2</sup> concentration, indicating a tunneling or hopping transport mechanism. The electronic transport could be modeled as taking place by thermally activated conduction along linkages or chains of *sp*<sup>2</sup> carbon atoms with variable range and variable orientation hopping, resulting in heterogeneity of conduction pathways through the sample. According to the model the annealing leads to chain ripening, a reduction in the activation energy for transport, and an increase in the electrical conductivity [10]. Annealing DLC films at 300°C was found to reduce the density of paramagnetic defects leading to a reduction in the dark conductivity and an increase in the photosensitivity [11]. The effect was attributed to the migration of hydrogen through the C–C network, leading to a better passivation of dangling bonds and a modification of the more weakly bonded *sp*<sup>2</sup> clusters with narrower local band gaps [11].

With the present understanding of the electrical properties of diamond-like carbon, it is apparent that neither DLC nor ta-C films are suitable to be used as semiconductor materials because of their relatively high disorder and defect density. Nevertheless, significant effort has been directed to study their usefulness for other electrical applications. One such potential application is the use of diamond-like carbon as a cathode material for thin film field emission displays.

## 2.2. Field emission

Flat panel field emission displays use sharp Mo or Si Spindt-type tips for emission of electrons for the excitation of phosphor screen pixels. The sharp tips are needed to obtain the high electric field required to extract the electrons from these materials which are characterized by a high work

function of about 5 eV. It was found that the threshold field for electron emission from diamond-like carbon films is much lower and can occur from flat films, thus not requiring the 1  $\mu$ m lithography for the preparation of sharp tips. The threshold field for emission in ta-C was found to reach values below 10 V/ $\mu$ m if the films contained a high fraction of about 80% of *sp*<sup>3</sup> bonds [12]. Addition of N to ta-C further reduced the threshold field, the maximum reduction to 5

V/ $\mu$ m being obtained in ta-C containing 1 at% nitrogen. Further increase in the nitrogen content reduced the *sp*<sup>3</sup> fraction in the films, reduced the bandgap, and increased the threshold voltage. Similar behavior has been observed in N-DLC films [13].

Diamond-like carbon coatings have also been found to improve the field emission characteristics of conventional Spindt type emission tips. Coating Si tips with 10-nm thick DLC reduced the turn-on voltage by 55%; however, the emission current still showed significant current fluctuation as observed with uncoated tips. Coating of Mo tips with DLC, alternating deposition and etching, or with N-DLC reduced the turn-on field by 45% and increased the emission current from 160 to 1520  $\mu$ A [14]. The emission current of the Mo-coated tips was much more stable than that of uncoated tips. Similar results have been reported for ta-C- or N-ta-C-coated Mo tips, but opposite results for Si-coated tips [15].

Field emission from unpatterned DLC or ta-C has been demonstrated for films deposited on a variety of substrates, such as Si wafer, Si wafers coated with different metals, SnO<sub>2</sub>, Corning 7059 glass, or conductive polymers [16]. Microstructuring the surface of a polymeric substrate by plasma treatment has led to a reduction of the emission threshold as compared to using the original substrate. The field emission properties of the diamond-like carbon can be influenced in some cases by its surface and contacts. The bottom contact surface appears to affect less the emission properties. It has been observed that 25-nm thick ta-C films deposited by filtered cathodic vacuum arc (FCVA) on *p*-type Si, *n*-type Si, SnO<sub>2</sub>, Au, Pb, Al, Mo, Cr, Cu had threshold fields for emission in the range of 5–15 V/ $\mu$ m without direct dependence on the work function of the back contact material [17]. However, similar films deposited on Ti and W had significantly higher threshold fields in the range 30–35 V/ $\mu$ m. This difference was attributed to the native oxide which was present between the back contact and the ta-C film in the latter case.

Furthermore, modification of the top surface of diamond-like carbon by plasma etching in H<sub>2</sub> or O<sub>2</sub> resulted in a reduction of the threshold field and increase in the emission current, in spite the fact that the surface did not roughen and even if the top 1–2 nm *sp*<sup>2</sup> layer was not completely removed by the etching [17]. It was shown also theoretically that the main barrier for electron emission from DLC deposited on Si is at the front surface [18].

As mentioned above, doping can improve the field emis-

sion properties of DLC or ta-C. Nitrogen decreases the emission barrier by increasing the bulk Fermi level and decreasing the work function, therefore generally improves the field emission from these films. Turn-on fields as low as  $4 \text{ V}/\mu\text{m}$  have been reported for N-DLC [13]. The effect of N incorporation in ta-C prepared by FVCA was found to enhance with increasing N incorporation [19]. At the highest N concentration the turn-on field was reduced to  $10 \text{ V}/\mu\text{m}$  in these films, and an average current density of  $0.1 \text{ mA}/\text{mm}^2$  was obtained at  $50 \text{ V}/\mu\text{m}$  assuming that the entire film surface was emitting. Sputtered carbon films containing 0.6 at% nitrogen have shown field emission currents of  $0.6 \text{ mA}/\text{mm}^2$  at an electrical field of  $5.8 \text{ V}/\mu\text{m}$  and a turn-on field of  $3.2 \text{ V}/\mu\text{m}$  [20].

Other elements besides N can affect the field emission properties of DLC or ta-C. It has been observed that the incorporation of boron into the PLD DLC films enhanced the electron field emission properties of the films [21]. AFM measurements revealed that the B-doped DLC contained clusters 80–90 nm in size. While the turn-on field for electron emission was lowered from  $E_{\text{ODLC}} = 9 \text{ V}/\mu\text{m}$  to  $E_{\text{OB DLC}} = 8 \text{ V}/\mu\text{m}$  by B doping, a much stronger effect was observed on the emission current density which increased from  $J_{\text{eDLC}} = 80 \mu\text{A}/\text{cm}^2$  to  $J_{\text{eB DLC}} = 2500 \mu\text{A}/\text{cm}^2$ . The increase in the emission current was attributed mainly to an increase of DLC conductivity as a result of the B doping [21]. Addition of Cs and its postdeposition oxidation has also been claimed to improve the field emission properties of carbon-nitrogen films [22].

The electron field emission properties of N-DLC films have been found to be improved by annealing at  $400^\circ\text{C}$ , the improvement increasing with increasing annealing time [23]. The specific mechanisms causing the improvement in this case are not clear since such an annealing causes changes in both the structure and composition of the hydrogenated films and also a reduction in film thickness due to loss of material. A similar effect of annealing was observed for N-DLC coated Si emitters [24]. The hydrogen loss occurring in N-DLC during annealing at temperatures above  $300^\circ\text{C}$  resulted in an increase in the conduction of the N-DLC films and caused a significant lowering of the turn-on voltage and increasing of the emission current of the silicon emitters. The effect was further enhanced by annealing at  $600^\circ\text{C}$ , presumably due to further improvement of the conduction through the N-DLC layer [24].

The easy electron emission from DLC or ta-C can be explained either assuming that the barrier to emission is very low or that very high field are involved. At the present stage there is no complete and common explanation of the observed field emission phenomena. The assumption of a very low emission barrier appears to have insufficient experimental evidence and the trend is, therefore, to explain the electron emission from diamond-like carbon films by the assumption of field enhancement. In some cases the field enhancement can be explained based on geometrical surface roughness of specific ta-C films. Thus, it has been observed

that both the current conduction through ta-C and the field emission current from ta-C are spatially inhomogeneous [10,25]. Similar nonuniformity of field emission has been reported for N-DLC films, where localized bright emission spots were observed when the average emission current density rose above  $8 \times 10^{-2} \text{ mA}/\text{cm}^2$  [13]. It was observed that the field emission occurs at individual sites, even from smooth ta-C or N-ta-C rather than uniformly across the entire surface at the turn-on field and the density of the emission sites can be as low as 10 sites/ $\text{cm}^2$  at  $30 \text{ V}/\mu\text{m}$  [25]. When the surface of N-ta-C roughened with the incorporation of 15% N in the cathodic arc deposited films the density of the emission sites increased to 350 sites/ $\text{cm}^2$  at  $30 \text{ V}/\mu\text{m}$ . Furthermore, intentional roughening of the films by deposition of ‘nanocralline’ films resulted in an enhancing of the emission site density to  $10^5$  sites/ $\text{cm}^2$  at  $10 \text{ V}/\mu\text{m}$  and up to 106 sites/ $\text{cm}^2$  at  $20 \text{ V}/\mu\text{m}$  [25].

However, field enhancement cannot be caused by geometrical effects in extremely smooth DLC films [18]. A recent theoretical model explains the easy electron field emission from diamond-like carbon as resulting from intrinsic field enhancement, independently of geometrical roughness. According to the model the emission occurs mainly from nanometer sized surface regions unterminated by hydrogen and characterized by a high electron affinity. Electric fields from the anode focus towards these unhydrogenated surface areas, the fields ending on negative charges in an underlying depletion layer. The resulting downwards band bending creates very large fields which cause Fowler-Nordheim emission [18]. Nevertheless, in spite of the different models explaining the field emission from diamond-like carbon films, a complete explanation of the experimental results does not yet exist.

The experimental and theoretical results obtained so far indicate that much more work has to be done before conventional, smooth diamond-like carbon films, DLC or taN or their doped varieties, can be used as thin-film field emission displays to replace displays based on Spindt tips. Furthermore, the question if such films will enable the same resolution as the one achieved with the patterned sharp tips is still open.

### 2.3. DLC as low- $k$ material

While diamond-like carbon materials do not seem to be useful for active electronic devices, they do have the potential to be used in passive applications. One such application is the potential use of the hydrogenated DLC as a low dielectric constant ( $k$ ) insulating material for ULSI interconnects. Low- $k$  materials are needed for the back end of the line (BEOL) interconnect structures of ULSI circuits to improve their performance. Diamond has a dielectric constant of 5.6, which is significantly higher than  $k = 4.0$  of the presently used  $\text{SiO}_2$  dielectric. However, it was found that, by adjusting the deposition conditions, it is possible to obtain DLC films with the dielectric constants in the range

2.7–3.8 and fluorinated DLC (FDLC) films with dielectric constants  $k < 2.8$  [26–28]. The integration of such films in the BEOL interconnect structures, however, imposes further requirements on the low- $k$  materials, such as low stresses, good adhesion and thermal stability at 400°C. It was found that, for the as-deposited DLC films, the relatively high intrinsic stresses (up to 800 MPa) decrease but also the thermal stability decreases with decreasing  $k$  values [26].

FDLC films with dielectric constants  $k < 2.8$  have been prepared having intrinsic stresses below 200 MPa. Both DLC and FDLC films can be stabilized by annealing in an inert ambiance and a first metallization level in a Cu-based damascene process with DLC as the dielectric has been demonstrated [28,29]. Such films become attractive candidates as a low- $k$  dielectric for the BEOL interconnect structure; however, further integration issues have yet to be addressed and solved.

#### 2.4. Nonvolatile storage

While the high density of electronic trap states has impeded the use of diamond-like carbon for electronic devices, it was recently suggested that these traps can be exploited as a means for producing nonvolatile digital information storage. Memory effects have been observed in the electrical characteristics of N-ta-C films deposited by FVCA [30]. These effects were characterized by a decrease in resistance and an increase in the capacitance of metal/N-ta-C/metal structures when a negative bias beyond a threshold was applied. The effects were reversible with the application of a positive bias larger than the same threshold. The authors described the conduction in the two resistance states by two mechanisms: electron hopping near the Fermi level in the high resistance state and Poole–Frenkel conduction just below the conduction band edge in the low resistance state. Defining the high and low resistance states as 0 and 1 binary states, these memory devices were characterized by write times of 100 ns, read times of 100 ns, and effective memory retention times approaching 1 year [30].

### 3. Optical properties and applications

#### 3.1. Optical properties

DLC films are typically transparent in the infrared, with the exception of the CH absorbing bands centered at about 2900  $\text{cm}^{-1}$ , weakly absorbing in the visible spectrum, and are increasingly absorbing with decreasing wave length in the UV. A wide range of optical gap values ( $E_{\text{opt}}$ ), spanning the range from 0.38 to 2.72, were reported for DLC films prepared under presumably similar conditions, indicating the dependence of the property on the deposition system. For otherwise similar deposition conditions  $E_{\text{opt}}$  of DLC was found to shrink strongly for films deposited above 250°C [2,31].

The index of refraction, both the real part  $n$  and imaginary

part  $k$ , and its spectroscopic variation has been found to be dependent on the preparation conditions and hydrogen content of the films. Its value at 632.8 nm can be adjusted from 1.7 to 2.4 by adjusting the deposition conditions. The index of refraction is also affected by the hydrogen content in the DLC films and generally increases with decreasing concentration of bound hydrogen as shown elsewhere [26,32]. It is, however, dependent on the concentration of bound hydrogen and not total hydrogen content in the film [33]. A higher index of refraction usually indicates DLC with stronger crosslinking, higher hardness, and better wear resistance [32].

#### 3.2. Optical applications

While ta-C has properties similar, some of them even superior to DLC, only DLC appears to have found practical applications so far. Due to its IR transparency, DLC can be used for optical applications, such as antireflective and scratch resistant wear protective coatings for IR optics (at a wavelengths of 8–13  $\mu\text{m}$ ) made of Ge, ZnS, ZnSe [34]. DLC is being used as a coating of germanium optics to protect the components against corrosion and rain impact damage. It can also be used to protect zinc sulfide infrared windows, when used in combination with a GeC intermediate bonding layer [35]. The GeC acts as an adhesion and as a stress relief layer. DLC can also be used to protect the surface of Al mirrors used in thermal imaging systems against deterioration in the environment. Protective DLC films can also improve the properties and lengthen the working lifetime of organic photoconductors [36]. The low deposition temperatures of DLC allows its use as a wear-protective layer on products made of plastic and is therefore used for protection against abrasion of sunglass lenses made of polycarbonate [37].

Besides their application as protective optical coatings, diamond-like carbon films can be used for fabrication of optical components. Diamond-like carbon can be easily patterned by etching in oxygen plasmas. Using anisotropic  $\text{O}_2$  RIE etching in combination with hard masks such as  $\text{SiO}_2$  or Al [38], patterns with well defined rectangular profiles can be obtained. In combination with the IR transparency of the films, this enables the recording of IR diffractive optical components with good control of surface and pattern quality.

Attenuated phase shift masks (APSM) are good candidates for achieving improved processing windows in DUV lithography. They require a single layer with partial transmission between 4 and 9% and thickness adjusted to produce a 180° phase shift at the edges to minimize diffraction effects [39]. DLC films are preferable to other absorber materials because they can be RIE etched to achieve good linewidth control and obtain 90° sidewall angles. The transmission through hydrogenated DLC can be tuned by adjusting the hydrogen content. Excellent UV stability at 248 nm was achieved for DLC films sputtered from a graphite target

in a Ar/hydrocarbon mixture on rf-biased substrates. In contrast, films deposited in the same system in absence of substrate bias deteriorated under UV exposure in air but not in nitrogen. This instability was attributed to adsorption of oxygen and its subsequent diffusion into the films. DLC based phase shift masks have been shown to produce an approximate 30% improvement in the depth of focus (DOF) as compared to a binary Cr mask. For small contacts around 200 nm, the DOF process window was improved as much as 100%. The smallest vias printed with DLC APSM were 150 nm in diameter [39].

The potential of using ta-C films for photovoltaic applications has also been demonstrated. ta-C films with  $sp^3$  fraction of 88% and N-ta-C were deposited by FCVA on *p*-type Si wafers with resistivities of 1–10  $\Omega$  cm. The N-ta-C films had a *n*-type character with the Fermi level 0.8 eV below the conduction band [40]. The optical absorption coefficient of the ta-C and N-ta-C films were sufficiently large to absorb a significant fraction of the solar energy. A photovoltaic effect was observed from Si/N-ta-C structures and the reverse current measured in Si/N-ta-C structures increased by three orders of magnitude when the structures were exposed to AM1 light. The values of short circuit current, open circuit voltage, and fill factor were 5.05 mA/cm<sup>2</sup>, 270 mV, and 0.263, respectively. Lower corresponding values were observed for undoped ta-C films [40].

#### 4. Summary

The amorphous structure of diamond-like carbon films enables the adjustment of their properties over large value ranges; however, their disordered structure leads to poor semiconducting properties. The films are being used as wear and corrosion protective coatings of magnetic mechanical, electrical and optical devices, and for fabrication of optical components. They have a strong potential for application in passive electrical devices.

A large effort is being invested in investigating the suitability of DLC or ta-C for use as active elements in electrical devices, such as field emission displays. However, such applications have yet to be demonstrated for the conventional diamond-like carbon films.

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