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Atmospheric pressure PECVD of fluorocarbon coatings from glow dielectric barrier discharges

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Keywords

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Summary

The deposition of fluorocarbon films from GDBDs fed with helium-hexafluoropropene (He-C₃F₆) and helium-octafluoropropane-hydrogen (He-C₃F₈-H₂) mixtures was investigated. The effect of several parameters was evaluated in order to clarify both the GDBD existence domain and film characteristics. He-C₃F₆ GDBDs allowed to obtain films with a F/C ratio of 1.5 and to achieve deposition rates up to 35 nm·min⁻¹. By changing the H₂ content in He-C₃F₈-H₂ fed GDBDs it was possible to vary the crosslinking degree, to tune the F/C ratio of the coatings and to change the deposition rate which was maximised for a H₂/C₃F₈ ratio close to 1. A spectroscopic investigation of the plasma phase is also presented.

Introduction

Low pressure plasma deposition of fluorocarbon films has been extensively studied in last decades.^[1-11] The main goal achieved has been the capability of tuning the chemical composition (i.e. the F/C ratio) and cross-linking degree of the deposits and, hence, tailoring their final properties such as dielectric constant, friction coefficient, hydrophobic/oleophobic character. The role of the monomer, of additives (e.g. oxygen or hydrogen), and the effect of several process parameters such as input power, pressure, discharge regime (i.e. continuous and modulated discharges) has been investigated and the film chemical composition and structure have been correlated to the plasma phase composition. Hydrogen addition to saturated fluorocarbon monomers (e.g. CF_4 , C_2F_6 and C_3F_8) allows the deposition of coatings with variable F/C ratio and crosslinking degree;^[1,3,7] less fluorinated and more crosslinked coatings can be produced by increasing H_2 content in the feed.

For example among the different fluorocarbon monomers utilised in low pressure plasmas, with hexafluoropropene (C_3F_6) a deposition rate up to $0.35 \mu\text{m}\cdot\text{min}^{-1}$ was obtained and XPS analyses showed that the coating was characterized by a F/C ratio of 1.5 and mainly consisted by similar amounts of CF_3 , CF_2 , CF and C-CF groups.^[9-11]

Studies on the Plasma Enhanced Chemical Vapour Deposition (PECVD) in Dielectric Barrier Discharges (DBDs) at atmospheric pressure utilizing fluorocarbon monomers have been already published by different authors both in glow (GDBD)^[12-14] and filamentary regime (FDBD).^[15-17]

One of the first investigations was reported by Yokoyama et al.^[12, 13] which utilized tetrafluoroethylene (C_2F_4) in mixture with high flow rates of helium in order to generate an Atmospheric Pressure Glow Discharge (APGD). An increase of the deposition rate up to approximately $2 \mu\text{m}\cdot\text{h}^{-1}$ was observed as a function of the monomer flow rate and of the discharge current, and the XPS analyses showed that the F/C ratio of the coating ranged between 1.4 and 1.7. Afterwards, Y. R. Prat et al.^[14] proposed the employment of He-fluorocarbon fed APGDs to modify the inner surface of polyvinylchloride (PVC) tubes for biomedical applications. If C_3F_6 was used as

reactive gas the deposited coatings were characterized by a F/C ratio of 1.46 and approximately equal amounts of CF, CF₂ and CF₃ as determined by XPS analyses. P. Vinogradov et al.^[16, 17] investigated the deposition of fluorocarbon coatings in filamentary DBDs fed by several Ar-fluorocarbon mixtures. In situ investigation of the plasma phase was performed. The effect of oxygen and hydrogen addition to the feed gas was evaluated on the deposition rate and film composition.

Despite the interesting results published, detailed and systematic investigations are needed in order to demonstrate that with atmospheric pressure DBDs it is really possible to tailor coatings properties which is one of the most important features of low pressure PECVD processes.

In the present work, GDBDs fed with He-C₃F₆ and He-C₃F₈-H₂ gas mixtures were utilized to deposit fluorocarbon thin films. The discharge operational window was accurately evaluated by means of electrical measurements and the effect of the monomer, i.e. saturated (C₃F₈) or unsaturated (C₃F₆) fluorocarbon compound, on coating features was clarified; furthermore the effect of hydrogen addition was investigated.

Experimental

The experimental apparatus consists of a parallel plate electrode (5 mm gap) system contained in a Plexiglas box.^[18] Each electrode (30 x 30 mm² area) is covered by a 0.635 mm thick Al₂O₃ plate (CoorsTek, 96% purity, 9.5 relative permittivity). The plasma is generated by applying an AC high voltage (up to 4 kV_{p-p}) in the frequency range 0.5 – 30 kHz. The power supply is composed of a variable frequency generator (TTi TG215), an audio-amplifier (Outline PA4006) and a high voltage transformer (Montoux).

The electrical characterization was performed with a digital oscilloscope (Tektronix TDS2014); the voltage applied to the electrodes was measured by means of a high voltage probe (Tektronix P6015A), while the current was evaluated by measuring with a voltage probe (Tektronix P2200) the voltage drop across the 50 Ω resistor in series with the ground electrode. The average power

density dissipated by the discharge was calculated as the integral over one cycle of the product of the applied voltage and the current divided by the period and the electrode area.

The gas flow rates were controlled by MKS electronic mass flow controllers (MFC) and the system pressure was monitored by means of a MKS 122 baratron. In order to avoid overpressure, the Plexiglas enclosure was slightly pumped with a rotary pump (Pfeiffer).

Glow discharges were fed with He-C₃F₆ and He-C₃F₈-H₂ gas mixtures (Air Liquide Helium C, Zentek C₃F₆ 99% purity, Zentek C₃F₈ 99+% purity). Deposition processes were performed at a He flow rate (ϕ_{He}) of 4 slm and a fluorocarbon concentration of 0.01 %, while the H₂-to-C₃F₈ flow rate ratio was varied from 0 to 2. The total gas pressure was equal to 10⁵ Pa. The frequency (f) was changed in the range 15-30 kHz and the input voltage (V_a) was kept constant at 2.8 kV_{p-p}.

Optical emission spectroscopy (OES) was performed by collecting the UV-Vis spectra (200 - 900 nm) with an Optical Multichannel Analyser (OMA) equipped with a monochromator (0.300 m focal length, imaging monochromator ACTON SP-300i, 1200 grooves/mm grating) and a CCD detector (SpectruMM™ 100B, Princeton Instruments).

The chemical composition of the coating was studied by Fourier Transform Infrared spectroscopy (FT-IR) analyses performed with a commercial Bruker Equinox 55 FT-IR interferometer. XPS analyses were carried out using a Theta Probe spectrometer (Thermo Electron Corporation) equipped with monochromatic Al K_α X-ray source (1486.6 eV) operated at a spot size of 400 μm (100 W power). Survey (0 – 1200 eV) and high resolution (C 1s, F 1s, O 1s and N 1s regions) spectra were recorded at a pass energy of 200 eV and 150 eV, respectively. All spectra were acquired at a take off angle of 37°. A flood gun was used to balance surface charging. The C 1s signal for CF₂ component, with a binding energy of 292.5 eV,^[19] was used as an internal standard for charging correction. Best fitting of the XPS high resolution C1s spectra was performed using Avantage Data Spectrum Processing software (Thermo Electron Corporation); the full-width at half maximum (FWHM) of each peak component was allowed to vary in 1.7 – 2.2 eV range but for components belonging to the same signal it was usually allowed to differ up to 0.2 eV.

Coatings wettability was evaluated by static water contact angle (WCA) using a Ramé-Hart manual goniometer (model A-100); contact angles were calculated by averaging the measurements on five 2 μl drops of double distilled water. Film thickness was evaluated on substrates partially masked during the deposition using an Alpha-Step® 500 KLA Tencor Surface profilometer, while film morphology was investigated by means of Scanning Electron Microscopy (SEM) analyses performed with a Cambridge Stereo Scan 240 (Cambridge Instruments).

Results and Discussion

The electrical characterization of the discharge allowed to evaluate the existence domain of a GDBD for C_3F_6 and C_3F_8 containing gas mixtures. For concentrations in the feed gas up to 0.01 % and 0.025 % for C_3F_6 and C_3F_8 respectively, a glow discharge was obtained at excitation frequencies higher than 15 kHz and applied voltages lower than 3.0 $\text{kV}_{\text{p-p}}$ since, under these conditions, a periodical discharge current signal composed by only one peak per half-cycle^[20] was observed; otherwise a filamentary discharge was obtained. The electrical diagnostics of He- C_3F_8 - H_2 fed glow DBD demonstrated that at the C_3F_8 concentration of 0.01 % utilized in this study, up to 0.02 % of hydrogen could be introduced in the feed (and therefore the $[\text{H}_2]/[\text{C}_3\text{F}_8]$ ratio was 2) without any evidence of microdischarge formation.

The effect of the excitation frequency in the range 15 - 30 kHz at 2.8 $\text{kV}_{\text{p-p}}$ was investigated in He- C_3F_6 fed GDBD. By increasing the frequency, a linear growth of the discharge average power density from 0.26 to 0.57 $\text{W}\cdot\text{cm}^{-2}$ and of the deposition rate from 19 to 34 $\text{nm}\cdot\text{min}^{-1}$ was detected. The normalized FT-IR absorption spectrum of the film deposited at 25 kHz from C_3F_6 is reported in figure 1. The main spectral feature is the broad band between 900 and 1400 cm^{-1} due to CF_x stretching vibrations.^[5-7, 9, 21-24] The signal is composed by a shoulder at 1350 cm^{-1} due to the CF, a peak at 1225 cm^{-1} and a shoulder at 1190 cm^{-1} associated to CF_2 asymmetric and symmetric stretching, respectively, and by a weak peak at 980 cm^{-1} due to CF_3 . The wide signal between 1650 and 1850 cm^{-1} can be ascribed to the C=C and C=O stretching vibrations.^[5-7, 9, 21-25] The C=O bonds

are due to O₂ and H₂O contaminations in the deposition chamber and/or to oxygen uptake after atmospheric exposure. The FT-IR spectra were slightly affected by frequency variation since only the shoulder due to CF₂ symmetric stretching and the C=O/C=C signal appeared to be slightly more pronounced at lower frequencies.

The F/C ratio of the deposited films, 1.5, was not affected by frequency variation while, in agreement with FT-IR, a slight increase of the oxygen concentration from 1.8 to 3.7 % was observed with XPS by decreasing the frequency from 30 to 15 kHz. As expected, the high resolution C 1s region was quite complex. Figure 2 shows the best fit of the C 1s peak of the coating obtained at 25 kHz. Five components were utilized for the curve-fitting: CF₃ (294.5 ± 0.2 eV), CF₂ (292.5 ± 0.2 eV), CF (290.1 ± 0.3 eV), C-CF and CF=C (CO) (288.0 ± 0.2 eV), C-C (285.0 ± 0.3 eV).^[3, 8, 9, 24, 26] The component at 288.0 eV can likely be due to C-CF groups, fluorinated-unsaturated groups (CF=C)^[26] and oxygenated components (C=O and C-O). The shoulder in the high binding energy region, centred at approximately 296.5 eV, is probably ascribed to a shake-up component of unsaturated carbons. The shape of C 1s signal was not affected by frequency variation and CF₂ was always the main contribution.

In agreement with XPS, WCA measurements did not show significant changes of surface wettability for different excitation frequency; a static WCA of 106 - 108° was observed for all frequencies.

SEM investigation showed that the coating surface was rough with some evidences of powder and globules formation at 15 kHz while at higher frequency (25 kHz), smooth surfaces were deposited, without significant evidence of powder formation.

The He-C₃F₆ fed GDBD was characterized by emissions from helium and fluorine atoms^[27] and CF₂ band in the spectral range 240 – 350 nm (A¹B₁ – X¹A₁ system).^[1, 2, 28, 29] A continuum, assigned by d'Agostino et al.^[11] to CF₂⁺, centred at approximately 290 nm, as well as the C₂ Swan band^[28] were detected. Several emissions from impurities, such as nitrogen, oxygen and water, were observed along with CO⁺ (first negative system) signals.^[27, 28] Figure 3 shows a typical emission

spectrum in the wavelength range 220 – 380 nm. By increasing the frequency all the emission signal intensities and the F/CF₂ normalised intensity ratio increased.

With He-C₃F₈-H₂ fed GDBD the effect of hydrogen addition to the feed gas was investigated by varying the [H₂]/[C₃F₈] ratio in the feed from 0 to 2, at 25 kHz and 2.8 kV_{p-p}.

The trend of deposition rate was in good agreement with the results obtained with low pressure plasmas fed with saturated fluorocarbons and hydrogen,^[1, 3, 7] in fact the deposition rate was extremely low (1 nm·min⁻¹) without hydrogen, then increased up to a maximum of 12 nm·min⁻¹ at [H₂]/[C₃F₈]= 1 and then decreased to approximately 2 nm·min⁻¹ at [H₂]/[C₃F₈] = 2 (Figure 4).

The infrared spectra of film deposited at various [H₂]/[C₃F₈] ratios in the feed are reported in Figure 1. By increasing the H₂ concentration in the feed the intensity of CF₃ stretching band (980 cm⁻¹) decreases and the shoulder due to CF vibration (1350 cm⁻¹) becomes more pronounced. The intense shift of the CF₂ stretching band position from 1225 cm⁻¹ to 1142 cm⁻¹ and the broadening of the overall CF_x stretching band can be ascribed to the reduction of fluorine content in the film and to the formation of a more disordered and crosslinked polymeric network.^[7] By increasing the H₂ content in the feed gas, the broad band between 1600 and 1850 cm⁻¹, due to C=O and C=C groups, increases and OH stretching absorption (3400 cm⁻¹) appears. For [H₂]/[C₃F₈] ratio higher than 0.5, a weak CH_x stretching band at approximately 2950 cm⁻¹ is observed.

By increasing the [H₂]/[C₃F₈] ratio in the feed gas from 0 to 2, the XPS fluorine concentration in the film decreased from 59 % to 35 %, carbon concentration increased from 39 % to 55 %, i.e. the F/C ratio passed from 1.5 to 0.6 (figure 4), and an increase of the oxygen atomic concentration was observed, since the O/C ratio passed from 0.04 without H₂ to 0.17 at a [H₂]/[C₃F₈] ratio of 2. Some nitrogen was also detected even though its atomic concentration was always lower than 1.2 %. The best fit of C 1s signals (figure 2), performed with the same procedure already described for He-C₃F₆ GDBDs, showed that by increasing the hydrogen in the feed gas, the component at (288.0 ± 0.4) eV increased, while the components at higher BE (CF₃ (294.5 ± 0.2 eV), CF₂ (292.5 ± 0.2 eV) and CF (290.1 ± 0.3 eV)) decreased. The peak at 288.0 eV, which as previously discussed is also due to C-

CF and therefore can be considered correlated with the surface crosslinking degree, increased from 25 to 55 % as a function of the $[H_2]$ in the feed, while the intensity of the peak associated to more fluorinated groups decreased.

In agreement with FT-IR and XPS data, the static WCA decreased from 111 to 88° as a function of the hydrogen content in the feed gas.

He-C₃F₈-H₂ fed GDBDs were characterized by the same spectral features already observed in He-C₃F₆ fed GDBD. However, as hydrogen was added to the feed gas, the CH emission ($A^2\Delta - X^2\Pi$) was detected at 4300 Å,^[28] while the C₂ Swan band disappeared. Reproducible trends were detected for F, H, CF₂ and CH emissions. In particular, with hydrogen addition to the feed, the intensities of H and CH increased, that of F was reduced while the intensity of CF₂ showed a maximum. A steep increase of H/F and CH/CF₂ intensity ratios was observed as a function of hydrogen concentration

The effect of the excitation frequency was investigated by varying the parameter under study between 15 and 30 kHz at a 2.8 kV_{p-p} for a $[H_2]/[C_3F_8]$ ratio of 0.5. As expected, by increasing the frequency, a linear growth of the deposition rate from 2.9 to 11.5 nm·min⁻¹ was observed. FT-IR analyses of deposited film showed a less pronounced absorption at 1700 cm⁻¹ (C=O and C=C stretching), a complete disappearance of the OH stretching band at 3400 cm⁻¹ and a variation of the CF_x stretching band shape and position by increasing the frequency. XPS analyses showed that the increase of the excitation frequency resulted in an increase of the F/C ratio from 1 to 1.2, while the O/C ratio was reduced from 0.15 to 0.05. These results were supported by WCA values that slightly increased with frequency. Finally, as observed with He-C₃F₆ fed GDBDs, SEM analyses of deposited films showed a quite rough surface of the coating with evidence of powder formation at 15 kHz while smooth and powder-free surfaces were obtained at higher frequency.

Conclusions

The experimental results presented in this work showed that it is possible to deposit fluorocarbon thin films by means of He-C₃F₆ and He-C₃F₈-H₂ fed atmospheric pressure GDBDs. The effect of

the monomer, i.e. saturated and unsaturated fluorocarbon, and of H₂ addition was investigated. It was demonstrated that it is possible to control the deposition process by varying the excitation frequency and the feed composition; frequency mainly affects the deposition rate, while feed composition allows to control the chemical composition of the coatings.

The electrical diagnostics of the discharge showed that the threshold concentration that allows to operate in a glow regime is quite low (0.01 % for C₃F₆ and 0.025 % for C₃F₈). In spite of this concentration limit, that strongly narrows the GDBD operational window, deposition processes were performed with an adequate process control.

As expected, the reactive gas plays a crucial role in the properties of deposited films. Deposition rates up to 35 nm·min⁻¹ were obtained with C₃F₆ as monomer, while with C₃F₈ the deposition rate was extremely low and increased with the addition of H₂. He-C₃F₆ fed GDBDs resulted in fluorocarbon films with a F/C ratio of 1.5; a significant evolution of the film composition was not observed as a function of excitation frequency, even though a slight increase of oxygen uptake was detected at low frequency. Smooth and powders-free coatings were deposited only at high frequency.

The trend of the deposition rate and the evolution of the chemical composition and crosslinking degree of deposited films in He-C₃F₈-H₂ fed GDBDs appeared to be in agreement with the results reported for fluoropolymer deposition in low pressure plasmas.^[1, 3, 7] In particular it was possible to tune the F/C ratio by varying the concentration of H₂ in the feed. H₂ admission was responsible for the increase of the deposition rate that was maximized for a fluorocarbon-to-hydrogen ratio close to 1. Reproducible trends were observed for the emission signals detected by OES; even though semi-quantitative hypotheses can not be performed, the steep increase of the CH/CF₂ and H/F intensity ratio as a function of H₂ content of the feed could be utilized for process control. Finally, the increase of the frequency resulted in a linear growth of the deposition rate, a slight increase of the F/C ratio, a decrease of the O/C ratio and a reduction of the roughness of the coatings.

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Figure captions

Figure 1. Normalized FT-IR spectra of film deposited in He-C₃F₆ fed GDBD ($f = 25$ kHz, $V_a = 2.8$ kV_{p-p}, [C₃F₆] = 0.01 %) and He-C₃F₈-H₂ fed GDBDs at different values of the [H₂]/[C₃F₈] ratio ($f = 25$ kHz, $V_a = 2.8$ kV_{p-p}, [C₃F₈] = 0.01 %, [H₂]/[C₃F₈] ratio = 0, 1, 2).

Figure 2. Best fit of high resolution C 1s spectra of fluorocarbon films deposited in He-C₃F₆ GDBD ($f = 25$ kHz, $V_a = 2.8$ kV_{p-p}, [C₃F₆] = 0.01 %) and in He-C₃F₈-H₂ GDBDs at different [H₂]/[C₃F₈] feed ratio ($f = 25$ kHz, $V_a = 2.8$ kV_{p-p}, [C₃F₈] = 0.01 %, [H₂]/[C₃F₈] ratio = 0, 1, 2). Experimental data are reported as black line, while the peaks originated by curve fitting are grey.

Figure 3. Emission spectrum of a He-C₃F₆ GDBD at $f = 25$ kHz, $V_a = 2.8$ kV_{p-p}, [C₃F₆] = 0.01 %.

Figure 4. Effect of [H₂]/[C₃F₈] ratio in the feed on deposition rate and F/C XPS ratio ($f = 25$ kHz, $V_a = 2.8$ kV_{p-p}, [H₂]/[C₃F₈] ratio = 0 - 2, $\phi_{He} = 4$ slm, $\phi_{C_3F_8} = 0.4$ sccm, $\phi_{H_2} = 0 - 0.8$ sccm).

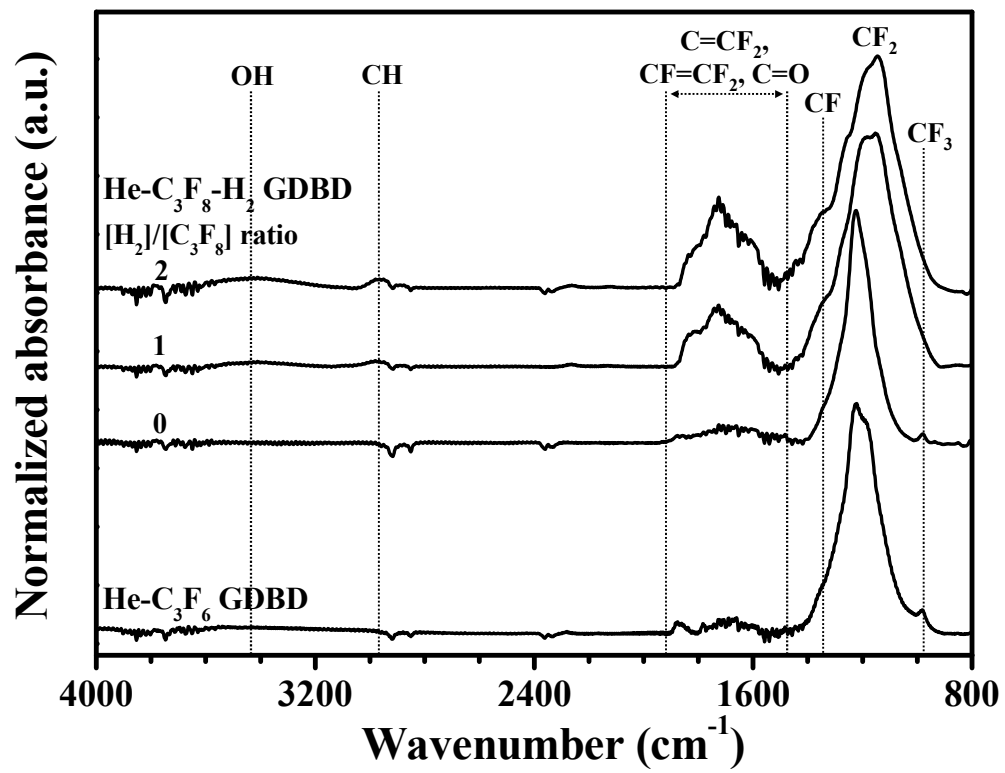


Figure 1

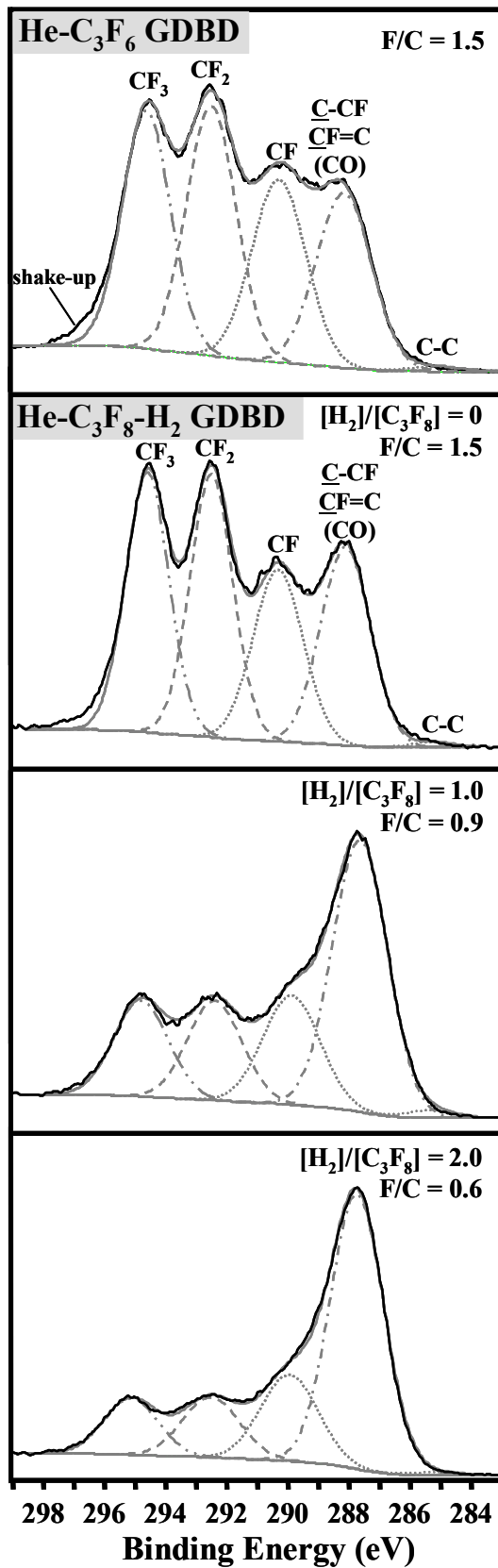


Figure 2

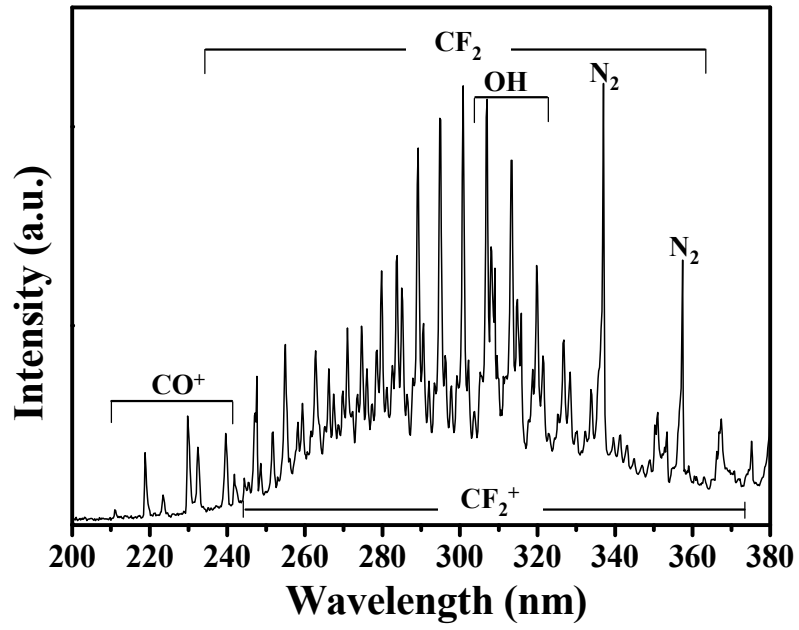


Figure 3

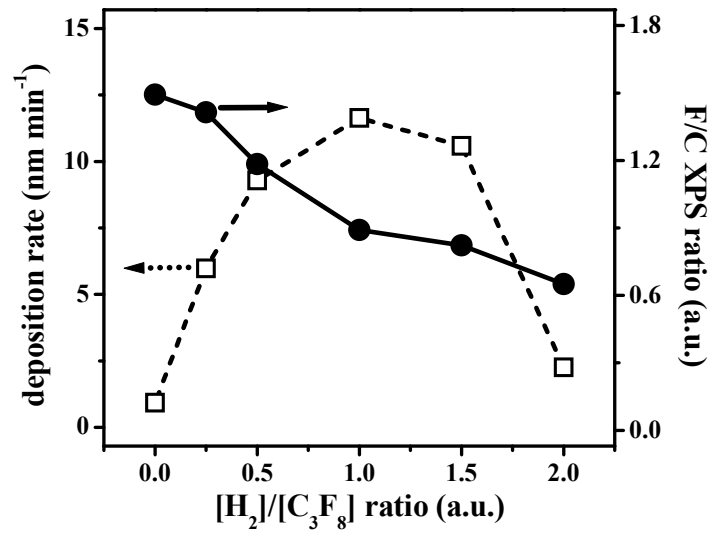


Figure 4

Text for the Table of Contents

Fluorocarbon thin films were deposited from He-C₃F₆ and He-C₃F₈-H₂ fed GDBDs. The effect of several parameters was studied in order to clarify both the discharge operational mode and coatings characteristics. A spectroscopic investigation of the main emitting species was also performed.