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PAPER

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Interpretation of artifacts in Fourier transform infrared spectra of atmospheric pressure dielectric barrier discharges: relationship with the plasma frequency between 300 Hz and 15 kHz

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Abstract

This article describes the occurrence of a phenomenon that is observed while recording mid-infrared ($4000\text{--}700\text{ cm}^{-1}$) absorption spectra of dielectric barrier discharges sustained at frequencies ranging from 300 Hz to 15 kHz. This phenomenon is observed as the presence of very sharp spikes in the spectrum, for which the wavenumber depends on both the high voltage frequency used to generate the discharge and the velocity of the moving mirror of the interferometer (which in turn determines the interferogram sampling frequency). While it is well known that the consumption of gas precursor within plasmas can be followed, we demonstrate that Fourier transform infrared spectroscopy also makes it possible to monitor frequencies and coupling of excitation mechanisms occurring in the plasma.

Keywords: dielectric barrier discharge, Fourier transform infrared spectroscopy, atmospheric pressure plasma, high-voltage plasma excitation frequency, interferogram sampling frequency

(Some figures may appear in colour only in the online journal)

1. Introduction

The dielectric barrier discharge (DBD) configuration [1, 2] that enables the creation of nonthermal plasmas at atmospheric pressure has gained increasing interest in the past years, with applications in surface functionalization [3],

synthesis of homogeneous or nanostructured thin films [4–8], removal UPR 8521, of pollutants [9], disinfection of medical tools [10], as well as tissue sterilization [11] and solar cells production [12] to name only a few. Despite the fact that DBDs are nowadays part of several industrial processes, the nature of the excited species that they contain and the mechanisms by which they interact with material surfaces still has to be understood. In this context, the chemical transformation

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of the active species in DBDs is a key point that requires new characterization methods to be developed.

Fourier transform infrared (FTIR) spectroscopy is a powerful tool to identify the chemical structure of molecules through the assignment of the vibrational frequencies of their chemical groups, either in the gas [13], liquid [14], or solid phases including thin layers [15, 16] and powders [17]. An interest has grown in recent years to extend the utility of FTIR spectrometry to analyze gaseous precursors in the plasma phase, while working in a DBD. This approach was shown to enable the analysis of the precursor consumption and dissociation in the plasma phase and the correlation of this information with the chemical composition of the plasma deposited thin film [18–21].

In the most common sampling configuration, the infrared beam is directed through the sample. In this situation, the infrared spectrum is calculated by ratioing the intensity of the beam that passes through the sample to that of the beam traveling through the entire spectrometer optical path in the absence of the sample [22]. In the present work, the aforementioned sampling configuration was used to characterize atmospheric plasmas, using an experimental setup previously described by Laroche *et al* [23]. For plasmas generated at high-voltage frequencies lower than 15 kHz, sharp spikes were observed in the infrared spectra at frequencies that depended on both the frequency of the high voltage used to sustain the plasma discharge and the interferogram sampling rate. Despite the fact that such spectral artifacts have already been observed by other groups working with similar plasma frequency conditions, their cause has not been assigned [21] or their presence was identified as a peak absorbance [24]. Knowing for which conditions and at which wavenumber the spike appears would help in experiment preparation to avoid overlap with infrared features of interest. In addition, understanding this peak origin would enable to use it as a tool to characterize the plasma as its wavenumber is shown to be determined by the plasma frequency. Therefore, it is essential to shed more light on this phenomenon and bring it to the attention of the plasma scientific community, as a limitation of FTIR for the study of plasmas, but also as a new method of plasma characterization. This article aims to understand and interpret the origin of this measurement artifact.

2. Methods

The plasma reactor in which the infrared studies were performed has been previously described by Brunet *et al* [6]. Briefly, the plasma reactor upper electrode was connected to the high voltage (HV) while the bottom electrode was grounded. Both electrodes were covered with an alumina plate acting as the dielectric barrier in the plasma reactor configuration. The HV was issued from a low frequency waveform generator (Agilent 33210) connected to an audio amplifier (Crest CC4000) linked to a voltage transformer (Boige et Vignal). The transformer frequencies ranged from 1 to 10 kHz with a voltage ranging between 1 and 15 kV. The gap between the electrodes was kept at 2 mm. The pressure within the reactor chamber was first reduced to 2×10^{-2} Torr to remove

impurities. Nitrogen was then introduced within the chamber to bring the reactor pressure back to 760 Torr. At this time, argon was introduced between the electrodes. The filling of the chamber with nitrogen followed by the injection of argon between the electrodes was done because nitrogen has 10 times higher breakdown voltage than argon. Hence an easy ignition is induced in between the electrodes, whilst nitrogen prohibits arc creation around the electrode [25].

The optical layout used to record mid-infrared absorption spectra in atmospheric plasmas has been described in detail elsewhere [23]. The infrared spectrometer was an Agilent Cary 670 equipped with a liquid nitrogen-cooled narrow-band mercury cadmium telluride (MCT) detector. Depending on the nature of the information that was targeted, between 1 and 256 scans were co-added, apodized using a medium Norton-Beer function, and Fourier transformed. The spectra were recorded between 700 and 4000 cm^{-1} at a resolution of 4 cm^{-1} . The background spectrum was recorded without any applied voltage between the electrodes. To help understanding the cause of the phenomenon of interest, the interferogram sampling rate was set either to 12.5 kHz, 25 kHz, or 37.5 kHz as determined by the optical velocity of the interferometer moving mirror and the HeNe laser beam wavenumber (ca. 15 800 cm^{-1}).

3. Results and discussion

Before trying to explain the appearance of spikes in the plasma spectra, it is worth mentioning some key observations. First, this observation is true whatever the breakdown voltage as observed by comparing different gases, which have breakdown voltage varying from 1.4 kV – Ar + NH₃, 2.7 kV – Ar to 9 kV for He²⁵). Thus, the presence of the spike is neither related to the voltage of the power supply nor to the plasma regime. Second, there is no observance of this peak when the plasma is on, but the infrared beam path directed aside from the plasma region. These two observations therefore eliminate any possibility of an electronically induced phenomenon. Third, this peak artifact could be observed without the infrared beam crossing the plasma region. Indeed, blocking the spectrometer infrared beam before its entrance in the plasma and only recording the signal from the emitted plasma light led to the recording of noise when a high number of scans (typically 128 or 256, which is usually used to record the FTIR spectra of plasmas) are co-added, while recording only one scan led to spectra with one or more discrete spikes. Of note, these spikes were observed at identical wavenumbers in various gas environments and plasma regimes (e.g. in argon, helium, argon + ammonia; results not shown), therefore indicating that their position is related to the plasma excitation frequency as opposed to the gas environment. Along with the other experimental observations, this points toward the fact that plasma infrared spectra are the result of the sum of the modulated signal originating from the spectrometer infrared source and the infrared signal coming from the plasma which is, in turn, modulated at the frequency of the HV that is used to sustain the discharge and not synchronized with the interferometer sampling process. Finally, the absorbance of these

Figure 1. Occurrence of the infrared artifact observed for plasma high voltage excitation frequencies of 4 (a), 5 (b), and 6 (c) kHz at a sampling rate of 25 kHz. The spectra are offset for clarity.

Figure 2. Occurrence of the infrared artifact observed at a plasma high voltage frequency of 2 kHz and interferogram sampling rates of 37.5 (a), 25 (b), and 12.5 (c) kHz. The spectra are offset for clarity.

spikes can be either positive (as exemplified by the spikes seen in figures 1(a) and (c)) or negative, as seen in figure 1(b). They can also have the shape of the first derivative of a spike going in the positive or negative direction. This observation leads to the deduction that the cause of the spike is out-of-phase with the corresponding Fourier frequency in the interferogram.

Figure 1 shows infrared spectra of argon plasmas at applied frequencies ranging from 4 to 6 kHz at a sampling rate of 25 kHz. As may be seen in this figure, a single spike can be observed in each of these spectra. The position of these peaks clearly depends on the applied voltage frequency used to sustain the plasma discharge and the ratio of their wavenumbers is identical to the ratio of the applied voltage frequencies.

The cause of these spikes must be some type of sinusoidal interference. The varying shape and direction of the spikes indicates that the phase of the interfering signal must not be correlated to the phase of the corresponding frequency in the interferogram.

As seen in figure 2, the frequency at which the artifact peak is observed also depends on the optical velocity of the interferometer and hence the interferogram sampling frequency, f_s . For plasmas generated with a voltage frequency of 2 kHz, the peak position is 840, 1263, and 2523 cm^{-1} for sampling frequencies of 37.5, 25, and 12.5 kHz, respectively. In this case, the ratio of the wavenumbers of the observed peak is proportional to the inverse of the high voltage frequency ratio.

Table 1. Comparison between the calculated and measured artifact wavenumbers.

Plasma excitation frequency (Hz)	Interferogram sampling frequency of 12.5 kHz		Interferogram sampling frequency of 25 kHz		Interferogram sampling frequency of 37.5 kHz	
	Calculated (cm ⁻¹)	Measured (cm ⁻¹)	Calculated (cm ⁻¹)	Measured (cm ⁻¹)	Calculated (cm ⁻¹)	Measured (cm ⁻¹)
100	126		63		42	
200	253		126		84	
300	379		190		126	
400	506		253		169	
500	632		316		211	
600	758		379		253	
700	885		442		295	
800	1011		506		337	
900	1138		569		379	
1000	1264		632		421	
2000	2528	2526	1264	1263	843	842
3000	3792	3793	1896	1895	1264	1263
4000	5057		2528	2528	1686	1685
5000	6321		3160	3160	2107	2105
6000	7585		3792	3792	2528	2528
7000	8849		4425	4422	2950	2949
8000	10113		5057	5056	3371	3371
9000	11377		5689	5686	3792	3789
10000	12642		6321	6319	4214	4212
11000	13906		6953	6950	4635	4634
12000	15170		7585		5057	5056
13000	16434		8217		5478	5476
14000	17698		8849		5899	5897
15000	18962		9481		6321	6320

Figure 3. Observation of two peak artifacts at a plasma high voltage frequency of 800 Hz and interferogram sampling frequency of 12.5 kHz.

Figure 4. Calculations of the fundamental (black curve) and first harmonic (red curve) artifact wavenumber at an interferogram sampling frequency of 25 kHz, showing the plasma high voltage frequencies at which the infrared artifacts can be observed in the mid-infrared region corresponding to the FTIR spectrometer effective optical range. (Blue) Only the fundamental component of the peak artifact is observed; (pink) only the first harmonic component of the peak artifact is observed; (green) both the fundamental and the first harmonic components of the peak artifacts are observed.

Table 2. Calculated values for peak artifact position (fundamental and 1st harmonic) for interferogram sampling frequencies.

Plasma excitation frequency (Hz)	Interferogram sampling frequency of 12.5 kHz		Interferogram sampling frequency of 25 kHz		Interferogram sampling frequency of 37.5 kHz	
	Calculated fundamental (cm ⁻¹)	Calculated harmonic (cm ⁻¹)	Calculated fundamental (cm ⁻¹)	Calculated harmonic (cm ⁻¹)	Calculated fundamental (cm ⁻¹)	Calculated harmonic (cm ⁻¹)
100	126	253	63	126	42	84
200	253	506	126	253	84	169
300	379	758	190	379	126	253
400	506	1011	253	506	169	337
500	632	1264	316	632	211	421
600	758	1517	379	758	253	506
700	885	1770	442	885	295	590
800	1011	2023	506	1011	337	674
900	1138	2275	569	1138	379	758
1000	1264	2528	632	1264	421	843
2000	2528	5057	1264	2528	843	1686
3000	3792	7585	1896	3792	1264	2528
4000	5057	10113	2528	5057	1686	3371
5000	6321	12642	3160	6321	2107	4214
6000	7585	15170	3792	7585	2528	5057
7000	8849	17698	4425	8849	2950	5899
8000	10113	20227	5057	10113	3371	6742
9000	11377	22755	5689	11377	3792	7585
10000	12642	25283	6321	12642	4214	8428
11000	13906	27812	6953	13906	4635	9271
12000	15170	30340	7585	15170	5057	10113
13000	16434	32868	8217	16434	5478	10956
14000	17698	35396	8849	17698	5899	11799
15000	18962	37925	9481	18962	6321	12642

Figure 5. Interferograms recorded after 1 (black) and 64 (red) scans for plasma excitation frequency set at 1 kHz and interferogram sampling frequency of 25 kHz.

Taken together, the data presented in figures 1 and 2 enable a very simple relationship to predict the wavenumber, w , where the spike will be observed to be established:

$$w = \frac{15\,800\text{ cm}^{-1} \times f_m\text{ (Hz)}}{f_s\text{ (Hz)}} \quad (1)$$

where f_m is the frequency of the high voltage used to sustain the plasma discharge, and f_s is the interferogram sampling frequency.

Table 1 reports the calculated and the measured wavenumbers of the artifact peak for different interferogram sampling frequencies and applied high voltage frequencies to sustain the plasma discharge. As may be seen, the peak artifact may be accurately predicted from equation (1).

Interestingly, some of the recorded spectra display two peak artifacts, with the higher wavenumber peak value being twice that of the lowest wavenumber feature, as seen in figure 3. It is therefore likely that the high wavenumber component is a harmonic of the fundamental peak observed at lower wavenumber. In these circumstances, it is also possible to calculate the wavenumber at which this harmonic contribution should be observed by simply multiplying the right term of equation (1) by two.

At this point, one could raise the question whether the presence of a single peak artifact in the recorded infrared spectrum necessarily means that the frequency of the associated phenomenon corresponds to the fundamental transition. The answer to this question is brought in figure 4, which correlates the spectrometer effective optical range (approx. between 700 and 4000 cm^{-1}) with the plasma HV frequency at a sampling frequency of 25 kHz. As may be seen in this figure, it is actually the harmonics that are seen up to a HV frequency of 1 kHz. On the other hand, both the fundamental and the harmonic component wavenumbers fall within the spectrometer effective optical range for HV frequencies ranging between ≈ 1 and 3.2 kHz, therefore leading to the observation of both the

fundamental and its harmonic. Finally, only the fundamental feature is observed at HV frequencies higher than 3.2 kHz.

All in all, this means that care should be taken while attempting to ascertain the frequency of the plasma HV modulation frequency from the wavenumber of the artifact peak in the case where only one peak is observed in the spectrum. Indeed, several FTIR measurements have to be performed at different sampling rate and plasma frequencies to correctly assign whether the artifact feature is a fundamental or a harmonic contribution. This is exemplified in table 2 where the calculated wavenumbers of both the fundamental and harmonic peak artifacts are reported as a function of the HV frequency for sampling rates of 12.5, 25, and 37.5 kHz.

Even though the wavenumbers at which the spikes should be observed can be precisely calculated, their origin still has to be identified. The description of the phenomenon illustrated above points toward the fact that the infrared light emitted by the plasma acts as a type of sinusoidal interference that combines with the sinusoidal components of the interferogram. Accordingly, if this hypothesis holds, the intensity of the bands related to the plasma light fluctuation should decrease with time as this signal would vary randomly from scan to scan whereas the amplitude of the interferogram increases linearly with the number of scans that are co-added. Thus, as noted above, the phase of the sinusoidal interference arising from the plasma should be independent of the corresponding phase of the sinusoidal components of the interferogram created by the scanning mirror of the interferometer. In terms of the origin of the phenomenon, the behavior can be explained since each half cycle of the voltage, the plasma switches on when the applied voltage reaches a certain threshold and switches off when the voltage applied to the gas decreases too much [26]. The spectrometer high-pass filter removes the low-frequency components, while the low-pass filter eliminates any high-frequency signal. Therefore, even if the plasma itself is a signal longer than a spike, which repeats every half-cycle

Figure 6. Occurrence of the infrared artifacts recorded at a sampling frequency of 25 kHz from a plasma generated at a HV frequency of 2 kHz, with an amplitude modulation set at a 100 Hz (10 ms) repetition frequency, during which the plasma is sustained for 5 ms, and then turned off for the following for 5 ms.

of the applied frequency, the detector signal looks like a series of spikes that appear each time the applied voltage reaches the plasma breakdown threshold.

Figure 5 shows the interferograms recorded after 1 scan and 64 scans at a sampling frequency of 25 kHz of an argon DBD generated at a high voltage frequency of 1 kHz. As may be seen in this figure, the interferogram recorded after a single scan nicely shows the superimposition of a series of spikes with the interferogram of the infrared source. The optical path difference between two successive interference peaks is approx. 0.8×10^{-3} cm. Considering that a maximal interference peak intensity should be observed at every mirror displacement of $\lambda/2$, the resulting wavelength value precisely matches with the prediction of equation (1) (with $\lambda = 1/\nu$). In addition, the interferogram recorded after co-adding 64 scans shows a clear decrease of the interference fringes observed after a single scan, therefore supporting the aforementioned hypothesis of the independence between the sinusoidal components of the phase generated by the pulsed infrared emission of the plasma and the phase of the sinusoidal components of the interferogram originating from the infrared source of the interferometer.

Figure 5 shows that the interference is neither a sine wave (in which case only the fundamental would be observed in the spectrum) or a square wave (in which case the fundamental and its odd harmonics would be seen). Instead it is better described as a shah function, III. The Fourier transform of the shah function is another shah function, with spikes at the fundamental frequency and both even and odd harmonics. The high harmonics are attenuated by the spectrometer's low pass filter so only the low-order harmonics are observed in the spectrum as shown in figure 3.

Figure 6 shows an interesting infrared spectrum recorded for a plasma generated at a HV frequency of 2 kHz with an

amplitude modulation (AM) set at a 100 Hz (10 ms) repetition frequency, during which the plasma is sustained for 5 ms, and then turned off for the following for 5 ms. As can be seen, the expected fundamental and 1st harmonic are observed at 1263 and 2525 cm^{-1} , respectively, (shown in black in figure 6) as predicted in table 2. However, these two peaks are surrounded by a series of two side-bands, for which the wavenumbers are indicated in red and blue. The differences of wavenumber between both main spectral artifacts and the peaks identified in red range between 60 and 71 cm^{-1} , which is comparable to the calculated value of the fundamental artifact component wavenumber that should be observed for a plasma with applied HV frequency of 100 Hz, shown in table 2. Furthermore, the wavenumber differences between the spectral components identified in red and blue from each side of the main artifact components range from 121 to 130 cm^{-1} , in close agreement with the expected value of the 1st harmonic artifact wavenumber when the plasma HV frequency is set as 100 Hz. These observations clearly put in evidence that FTIR spectroscopy can not only be used to observe plasma frequency modulation, but also to monitor the coupling between the principal plasma frequency and the plasma modulation frequency.

4. Conclusions

This study has shed more light on the origin of spectral artifact components which are observed while recording the FT mid-infrared spectra of dielectric barrier discharges generated at high voltage frequencies ranging from 300 Hz to 15 kHz. The results presented therein clearly demonstrated that the wavenumber of these spikes depend on both the high voltage frequency used to generate the plasma and the interferogram sampling frequency that is selected to record the FTIR spectra.

In addition, the FTIR spectra of plasmas submitted to a frequency modulation also display peak artifacts which originate from the interaction of this modulation with the plasma high voltage frequency. Therefore, in addition to enabling following gas precursor consumption within plasmas, FTIR spectrometry also makes it possible to monitor frequencies of excitation mechanisms occurring in the plasma. Future works will focus on the use of these infrared artifacts to quantify the coupling in plasmas generated using multiple frequency conditions.

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