Journal of Physics: Conference Series **591** (2015) 012063 doi:10.1088/1742-6596/591/1/012063

Optical and electrical characterization of C_3H_6O/Ar glow discharge

S. Villalobos[1, 2], F. Castillo[1], O. Flores[1], P.G. Reyes[3], H. Martínez[1],

[1] Laboratorio de Espectroscopia, Instituto de Ciencias Físicas, Universidad Nacional Autónoma de México, Apartado Postal 48-3, 62251 Cuernavaca, Morelos, México
[2]Facultad de Ingeniería, Universidad Nacional Autónoma de México, D.F., México
[3]Facultad de Ciencias, Universidad Autónoma del Estado de México, Toluca, México
ciro@fis.unam.mx

Abstract. A low pressure glow discharge apparatus was used to produce a glow discharge of C_3H_6O/Ar mixture at a total pressure of 2.0 Torr. The emission spectra were measured in the wavelength range of 200 to 1100 nm. The principal species observed were Ar I, C_6H_4O, C_6H_5, CHO, CH_3O, CO_2, CO, H_2O, and H_2. The electron temperature and ion density have been measured using a double Langmuir probe, and the electron temperature and ion density were found in the order of 10 eV and 10^{10} cm^{-3}.

I. - Introduction

Recently, numerous experimental and theoretical works have been conducted to study of plasmas that contains hydrocarbons. This interest was mistreated by the advance of technologies of synthesis of new materials with unique mechanical, electrical, and optical properties [1–3], and also by investigation of processes that occur in space [4], nuclear reactors [5], in detoxification of gas combustion products [6], and in synthesis of composite hydrocarbons [7]. For optimization of available plasma-chemical synthesis technologies and development of new ones, it is necessary to study plasma-chemical reactions and kinetics of nonequilibrium plasma containing hydrocarbons, their fragmentation, and monitor changes in chemical plasma composition.
Plasma is a state that is far from thermodynamic equilibrium; for this reason it is not easy to model chemical processes that occur in gas phase and on adjacent surfaces. Therefore, for verifying the models and results of calculating the processes in plasma, it must be headed to measure its composition immediately during the process (in situ). The situation is complicated by the fact that kinetics and properties of any plasma (laboratory, technological, or natural plasma) are governed by products of stable molecule decay, in particular, by radicals; meanwhile, there are few means for their diagnostics. Radicals and other fragments of molecules in plasma containing hydrocarbons were most frequently studied using optical emission spectroscopy (OES) [8], laser-induced fluorescence (LIF) [9], mass spectroscopy [10], and absorption spectroscopy [2, 11].

Generally, it is not easy to measure the radical concentration in plasma [12]. Direct measurements by (OES) require exact knowledge of electron density and energy along the observation region because, as a rule, only small part of electrons from the high-energy distribution function tail participates in excitation process. The LIF method requires complicated equipment, availability of proper energy levels filled with visible radiation from ground or excited states, and which is most important; the method makes it possible to measure only relative concentrations of radicals. Concerning the absolute values, their usage requires labor-intensive calibrations. Methods based on photon absorption in the visible, IR, and microwave regions were used for measuring absolute concentrations, of CH_3 [13], CH [14], and OH [15]. However, the most frequent method used for measuring radicals and other easily reacting plasma components was mass spectrometry [10].
In the present work, the goal was to explore the OES and to carry out measurements of electron temperature, and ion density in a glow discharge of Ar and C_3H_6O mixture at total pressure of 2.0 Torr.

II. – Experimental setup

The experimental apparatus and technique used to measure the AC plasma emission spectrum is identical to that described in previous work [16, 17]. A brief description is reported here. The system consists of two stainless steel circular plane electrodes, 3 mm thick and 30 mm in diameter. The electrodes are positioned at the centre of the reaction chamber with 10 mm gap spacing. The C_3H_6O and Ar were injected into the reaction chamber through the bottom flange. The same connection was used for the pressure sensor (MKS, Type 947 convection enhanced Pirani). An AC discharge of 60 Hz was used to generate the C_3H_6O-Ar plasma at a pressure of 2.0 Torr. An ultra pure Ar gas and C_3H_6O were used in the course of the measurements. The discharge power supply was maintained at an output of 450 Volts and a current of 3.5 mA (1.6 W). The vacuum chamber was purged with Ar gas several times at a pressure of 1.0 Torr in order to remove the background gas. In order to monitor the plasma discharge, in front of the lateral plane of the electrodes was located an optical fibber (Solarisation-resistant UV and fibber diameter size of 400 μm) that was connected to the entrance aperture of a high-resolution Ocean Optics Inc. Spectrometer Model HR2000CG-UV-NIR. The inlet and outlet slits were 5 μm wide. The data were obtained in a single accumulation of 100 s integration times. The fibber optics assembly was positioned to collect the emission light appearing on the C_3H_6O/Ar plasma. The wavelength accuracy of the spectrometer was calibrated using an Ar Source (Ocean Optics Inc.).
The double Langmuir probe employed in this work consists of a 0.25-mm-radius tungsten wire. The probe was located inside a capillary glass. The tips were extended 2.0 mm beyond the capillary glass with a gap between them of 2 mm [18]. The probe was aligned parallel to the electrodes of the discharge. The applied voltage to the probe was manually scanned from +30 V to -30 V and *vice versa* by a regulated DC power supply. The probe current was monitored by a multimeter. The scanning time for each *I-V* curve was 5-10 minutes. The final *I-V* curve obtained was the result of an average of 6 data scans, from this final curve the electron temperature (T_e) and ion density (n_i) were calculated.

III. - Results

To investigate the plasma properties, the plasma emission spectra were obtained with spectrometer (Ocean Optics) with an optical fibber though the Pyrex glass window. The measurement conditions, such as the integration time of the spectrometer and the position of the optical fibber, were fixed for every measurement.

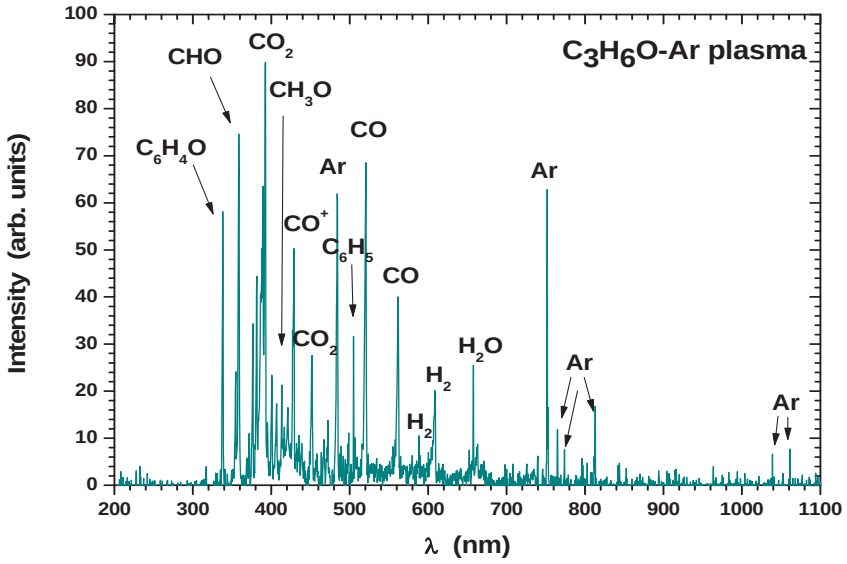

Fig. 1 The OES of C_3H_6O-Ar plasma at 2.0 Torr

A typical OES measurement of glow discharge C_3H_6O-Ar mixture at a pressure of 2.0 Torr is displays in Figure 1, showing the intensities of all observed emission bands and lines. This allowed the analysis of the most luminous area, which corresponds at the centre of discharge. Only the most intense spectral lines and bands of the plasma within the 200-1100 nm range are quoted. The principal species observed were: C_6H_4O at 338.11 nm; CHO at 358.75 nm; CO_2 at 391.2, 452.7 nm; CH_3O at 420.5 nm ; CO^+ at 427.43 nm; C_6H_5 at 504.8 nm; CO at 519.82 and 561.02 nm; H_2 at 588.82 and 608.08 nm; and Ar at 484.78, 751.46, 763.51, 771.37, 1047.01 and 1067.35 nm.

From the measured experimental current-voltage characteristic curve [18] obtained by the double Langmuir probe at a pressure of 2.0 Torr of glow discharge C_3H_6O-Ar mixture, it is possible to get electron temperature (T_e) and ion density (n_i). The T_e and n_i were obtained fitting the theoretical curve to the experimental data. With a correlation coefficient of 0.98, the theoretical curve fit very well to the measured data. That procedure gives an electron temperature of T_e = (7.31 ± 0.88) eV and ion density of ni = (1.28 ± 0.1) x 10^{10} cm^{-3}. Error in the T_e and n_i were discussed in reference [19] and the overall variations in T_e and n_i were found to be 10% and 12% respectively.

In order to analyse the material deposited (MD) on the electrodes, this was collected by mechanical scraping Figure 2, displays Scanning Electron Microscopy (SEM) of the MD on the electrode during 50 h. Overview of the film (knackered) deposited on the electrodes and placed in a graphite film. To determine the surface contact, observations were made in the light microscope. It found that flat faces were in contact with the electrode of copper (Cu), and the rougher faces are exposed to the plasma. It behavior maybe explained on basis the growing of material particles as it's were deposited in the electrode.

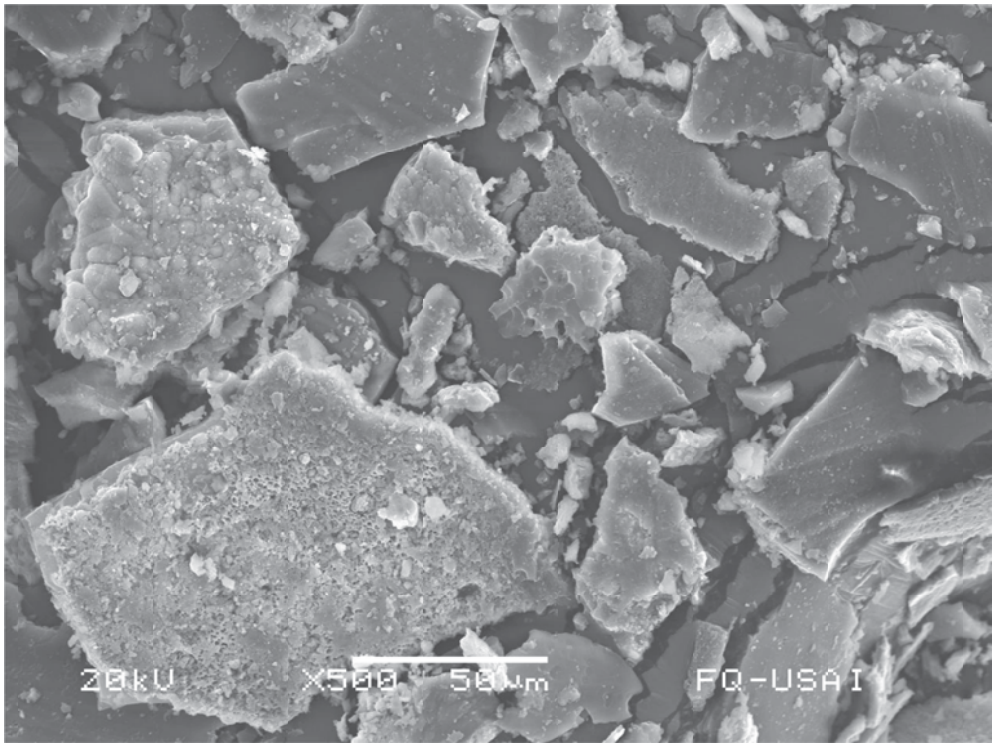

Fig. 2 SEM of the material deposited on the electrode.

Different textures of the surfaces of the powders have been observed, a smooth and one rough with granular appearance. The rough surface corresponding to the shelf and it has a granular structure, which it could be formed from the particles generated in the plasma and deposited on the surface of Cu. The particles have a size of 1.3 ± 0.6 microns. From the analysis by EDX of the material deposited can identify the presence of C (92.41%), and O (6.5%). With traces of Si (0.22%), and Cu (0.87%), elements which should be contaminants from the Cu substrate.

IV. - Conclusions

The main species observed in a glow discharge of C_3H_6O-Ar plasma were the molecular bands of CO_2, CHO, CH_3O, CO, C_6H_4O, C_6H_5 and Ar atomic lines. Likewise, by the technique of double Langmuir probe electron temperature and plasma ion density were determined. T_e = (7.31 ± 0.88) eV and n_i = (1.28 ± 0.1) x 10^{10} cm^{-3}. The deposit is mainly composed mostly of carbon and oxygen in less proportion. The processes contribute more acetone to the environment than natural processes conning from of hydrocarbon derivatives (ketones) by its presence in natural (plants, trees, volcanic gases and forest fires) as well as in the exhaust gas of automobiles, smoke snuff and landfill. So, a more complete study of the films formed on the electrodes, give us more information about the processes that occur and that give rise to these films.

Acknowledges

The authors are grateful to H.H. Hinojosa and J. Rangel for technical assistance. This research was partially sponsored by DGAPA IN101613, CONACyT 128714.

References

1. - L. Mechold, J. Ropcke, X. Duten, and A. Rousseau, Plasma Source. Sci. Technol. 10, 52 (2001).
2. - F. Hempel, P.B. Davies, D. Loffhagen, L. Mechold, and J. Ropcke, Plasma Sourc. Sci. Technol. 12, S98 (2003).
3. - N. Mutsukura, Plasma Chem. Plasma Process. 21, 265 (2001).
4.- P. Coll, D. Coscia, M.C. Gazeau, E. De Vanssay, J.C. Guillemin, and F. Raulin, Adv. Space Res. 16, 93 (1995).
5.- F.L. Tabares, D. Tafalla, I. Tanarro, V.J. Herrero, A. Islyaikin, and C. Maffiotte, Plasma Phys. Control. Fusion 44, L1 (2002).
6.- B.M. Penetrante, M.C. Hsiao, J.N. Bardsley, B.T. Merritt, G.E. Vogtlin, A. Kuthi, C.P. Burkhart, and J.R. Bayless, Plasma Sourc. Sci. Technol. 6, 251 (1997).
7.- M. Kareev, M. Sablier, and T. Fujii, J. Phys. Chem. 104, 7218 (2000).
8.- U. Fantz, Plasma Sourc. Sci. Technol. 15 S137 (2006).
9.- L. Magne, S. Pasquiers, V. Edon, F. Jorand, C. Postel, and J. Amorin, J. Phys. D: Appl. Phys. 38, 3446 (2005).
10.- P. Kae-Nune, J. Perrin, J. Guillon, and J. Jolly, Plasma Sourc. Sci. Technol. 4, 250 (1995).
11.- A.K. Ngai, S.T. Persijn, F.J. Harren, H. Verbraak, and H. Linnartz, Appl. Phys. Lett. 90, 081109 (2007).
12.- D.C. Schram, Plasma Sourc. Sci. Techno. 18, 014003 (2009).
13.- A. Welzel, A. Rousseau, P.B. Davies, and J. Ropcke, J. Phys.: Conf. Ser., 86, 012012, (2007).
14.- R. Engeln, K.G. Letourneur, M.G. Boogaarts, M.C. Van de Sanden, and D.C. Schram, Chem. Phys. Lett. 310, 405 (1999).
15.- J. Ropcke, G. Lombardi, A. Rousseau, and P.B. Davies, Plasma Sourc. Sci. Techno. 15, S148 (2006).
16.- H. Martínez, O. Flores, B. Campillo, A. Gomez, L. Salazar, and J.C. Poveda, Radiation Effets & Defects in Solide 167, 582 (2012).
17.- G. García-Cosio, H. Martínez, M. Calixto-Rodriguez, A. Gomez, J. of Quantitative Spectroscopy & Radiative Transfer 112, 2787 (2011).
18. - J.D. Swift and J.R. Schwar, Electric Probes for Plasma Diagnostics, Elsevier, New York, (1991)
19.- L. Salazar-Flores, H. Martinez, A. Guerrero, P.G. Reyes, J. of Physical Science and Application 2, 283 (2012).