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To cite this article: Qianghua YUAN *et al* 2019 *Plasma Sci. Technol.* **21** 025402

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OES diagnostic of radicals in 33 MHz radio-frequency Ar/C₂H₅OH atmospheric pressure plasma jet

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Received 26 April 2018, revised 16 October 2018

Accepted for publication 26 October 2018

Published 28 November 2018



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Abstract

Ar/C₂H₅OH plasma jet is generated at atmospheric pressure by 33 MHz radio-frequency power source. This RF excitation frequencies which are higher than 13.56 MHz had rarely been used in atmospheric pressure plasma. The plasma characteristics of ethanol are investigated. The introduction of ethanol leads to the generation of four excited carbonaceous species C, CN, CH and C₂ in plasma, respectively. Optical emission intensities of four carbonaceous species were strengthened with ethanol content increasing in the range of 0–4600 ppm. The ethanol content increase results in all the Ar spectra lines decrease. The reason is that the electron temperature decreases when ethanol content is high. The emission intensity ratios of C/C₂, CN/C₂ and CH/C₂ decrease with the increase of ethanol content, showing that the relative amount of C₂ is increasing by increasing the ethanol flow. The emission intensity ratios of excited species did not change much with the increase of RF power in stable discharge mode.

Keywords: atmospheric pressure plasma, optical emission spectroscopy, radical analysis

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

1. Introduction

Nonthermal atmospheric pressure plasma jets (APPJs) attracted a lot of attention because of technological applications in different fields, such as nanotechnology [1–4], biomedicine [5–8], polymer treatment [9, 10], the deposition of thin films [11, 12]. To APPJ, the plasma plumes are generated in air rather than in confined discharge gaps, thus they can be used to treat three-dimensional (3D) complex geometries without the space constraint. They will not harm the heat sensitive materials because the gas temperature of nonthermal plasma is low enough, but they contain many radicals to treat materials effectively.

In recent years, carbon nanomaterials are being considered for nano-electronics, sensors, and nano-probes because their electronic and mechanical properties are good. In addition, carbon nanotubes, spherical carbon onions

also are the focus because they have superior lubrication properties. Consequently, the preparations of various carbon nanomaterials have become research hotspot. The plasma-enhanced chemical vapor deposition (PECVD) and arc discharge are common methods [13–17]. Single-walled carbon nanotubes have been produced using PECVD method [13]. Fullerene-related carbon nanostructures have been synthesized by an arc-in-liquid system, and the alcohols, alkanes and aromatics were used as a carbon source [16]. The carbon nanoparticles can be obtained in the form of floating powder on the water surface and followed an arc discharge between two graphite electrodes which were submerged in water [17]. However, the PECVD need high energy consumption or expensive equipment, and arc discharge cannot be applied to the processing of heat sensitive materials.

APPJs have advantages because of their low gas temperature and energy consumption. Different kinds of APPJs

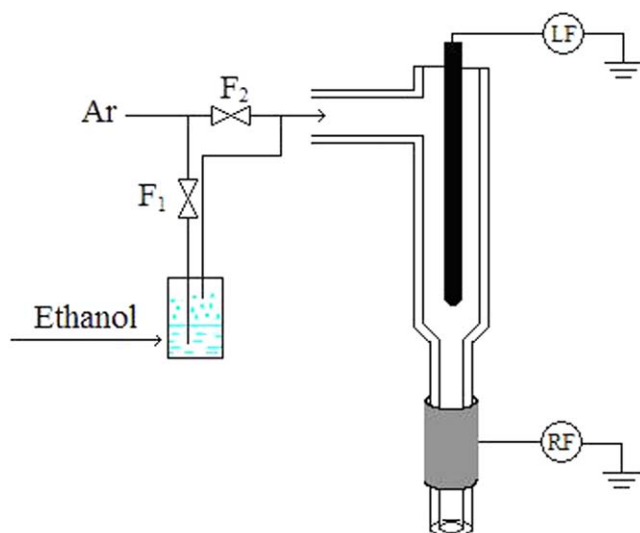


Figure 1. Scheme of the experimental setup of Ar/C₂H₅OH plasma jet.

generated with sinusoidal excitation at kilohertz, megahertz, microwave, and with high voltage submicrosecond pulse were experimentally studied [18–22]. APPJs are also used in the depositing of soft polymerlike hydrogenated amorphous carbon films using Ar/C₂H₂ mixtures [12]. Ethanol (C₂H₅OH) is rich in carbon and is very cheap compared with various carbonaceous gases. If kinds of carbon nanomaterial can be synthesized by using ethanol as carbon-based molecules conveniently and cost effectively with APPJs method, it will be a practical application. Therefore, the characteristic of ethanol plasma needs to study at atmospheric pressure. To plasma assisted chemical reactions, it is the crucial step in plasma processing to form reactive neutral and radicals. In this work, we studied the excited species in Ar/C₂H₅OH plasma using optical emission spectroscopy (OES). The effect of ethanol content and RF power on the discharge chemistry process was investigated.

2. Experimental setup

The RF plasma discharge, especially with higher frequency (such as 33 MHz), is difficult to ignite at atmospheric pressure [23–25]. However, it is unhelpful to obtain the uniform RF glow plasma because the Townsend breakdown threshold values is higher, so the glow discharge tend to change into a filamentary arc after breakdown under a relatively large electric field [26]. In order to reduce the ignition power, the plasma device is equipped with a low-frequency (LF) ignition device. In this work, the single RF is ignited at input power of 95 W, but the RF ignition power is 38 W when adding a LF ignition device. The schematic of the atmospheric pressure Ar/C₂H₅OH plasma jet is shown in figure 1. The details are described in our previous work as [22]. The inner and outer diameters of quartz syringe nozzle are 3 mm and 5 mm, respectively. The central copper pin electrode (2 mm diameter) is driven by a 50 kHz LF power source (CTP-2000K).

Except for the head, the copper pin is wrapped by a dielectric material (quartz glass). In this experiment, the Ar/C₂H₅OH mixture discharges at atmospheric pressure. The discharged plasma belongs to low temperature plasma, so the temperature of electron is not very high, and the electron bombarding energy is very low. Under low bombarding energy, the second electron of Cu is easy to be excited because Cu is metal. But to quartz glass dielectric, the electron is bound to nucleus, so the second electron is hard to be excited under low bombarding energy. The uncovered metal electrode can reduce the discharge voltage significantly compared with that covered with dielectric material completely. The emission lines of Cu can be observed with larger RF power if the copper pin is not covered by dielectric material. In order to eliminate the lines of Cu, the copper pin except for the head with dielectric material is necessary. If the head of copper pin also is wrapped by a dielectric material completely, the discharge is difficult because of lack of secondary electron emission at low temperature plasma. The copper ring electrode covered around the outside of the quartz syringe is driven by a 33 MHz RF power source (RSGK100). There are very few reports of APPJ which were generated by RF power source whose radio frequency is larger than 13.56 MHz. So, the 33 MHz was chosen to study. A spectrometer (Avaspec-2048-8) was used to collect the optical emission spectra. A bubbling system with ethanol (99.7%) was used to control the content of ethanol steam in the mixture.

According to Antoine's equation [27]

$$\log P = A - B/(T + C), \quad (1)$$

where P (saturated vapor pressure) is mmHg, and T (temperature) is Celsius, A , B and C are Antoine constants. The unit of P usually is described by kPa, and the unit of T is K. So in this paper, the mmHg unit of P is transformed for kPa (1 mmHg = 0.133 322 kPa). At the same time, the Celsius unit of T is transformed for K (1 °C = 274.15 K). So P is saturated vapor pressure (kPa), and T is temperature (K), A , B and C are Antoine constants (16.675 83, 3674.491, -46.702) in this paper. At room temperature (293 K), the saturated ethanol vapor pressure is 58.7×10^{-3} bar. The partial pressure of ethanol vapor in the gas mixtures is

$$P_{\text{C}_2\text{H}_5\text{OH}} = 0.0587 \times F_1/(F_1 + F_2)(\text{bar}). \quad (2)$$

The experiment condition was 1 atm, so the content of ethanol could be obtained from its partial pressure. The total flow ($F_1 + F_2$) of Ar was kept constant at 4 slm (standard liters per minute) in all measurements. The content of ethanol was varied from 0 to 4600 ppm by changing the gas flow passing through the bubbler. At first, the plasma was ignited by LF power source, then turned on the RF power source and following increase the RF power. The applied power was kept at 70 W (the incident power: 102 W, the reflect power: 32 W) for the different ethanol content, then the applied power was varied from 70 to 120 W. When the discharge can be maintained by the RF power source, then the LF power source was turned off. All measurements are conducted with single 33 MHz RF plasma. The LF power source is only used to assist ignition in this research.

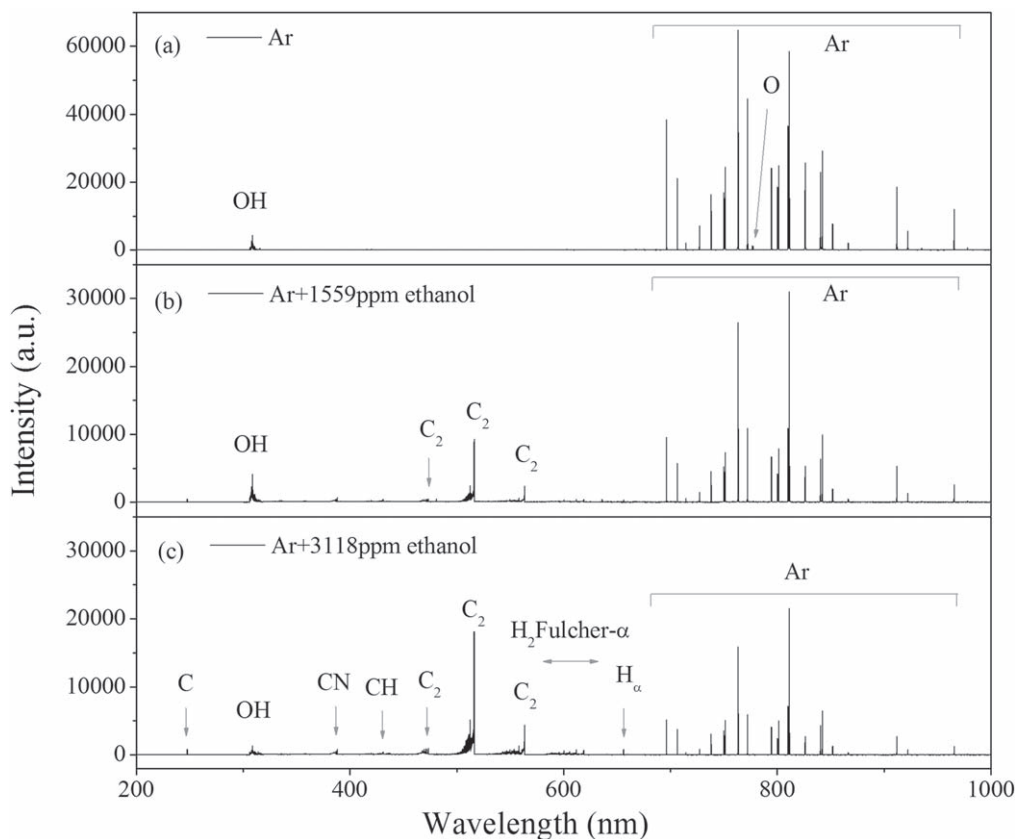


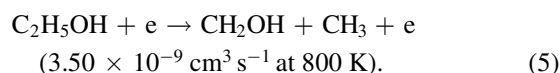
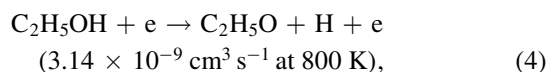
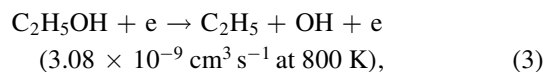
Figure 2. Optical emission spectra of the plasma jet at different contents of ethanol in Ar, the applied power was 70 W and the total was 4 slm.

3. Results and discussion

Regarding the experimental characterization of APPJs, OES is a common technique to observe excited species in plasma discharge. In the experiment, the ethanol (99.7%) precursor was carried by the Ar (99.999%). Before ignition, the mixture of Ar/C₂H₅OH flowed into the quartz for five minutes in order to dispel the interference of air. When plasma discharge, the main content is Ar/C₂H₅OH mixture. Because the content of N₂ was little, so they almost did not appear in optical emission spectra. In figure 2, optical emission spectra of the plasma jet at different contents of ethanol are presented. Adding ethanol to the feed gas, some spectral line and bands, such as C, CN, CH and C₂ were observed in figure 2. The Balmer series (656.3 nm) of H lines, and the Fulcher band system (580–650 nm) of H₂ also were observed, because of the dissociation of hydrogenous species and the recombination of H. The N₂ (337.1 nm) and O (777.2 nm) were observed in pure Ar discharge or adding a few ethanol. The N₂ (337.1 nm) and O (777.2 nm) emission became weaker and weaker when more and more ethanol was added, which means that the dissociation of ethanol is easier than excitation of N₂ and dissociation of O₂. At present, there has no generally accepted kinetic mechanism for modeling plasma kinetics in Ar/C₂H₅OH mixture. A kinetic mechanism proposed by Levko *et al* [28] has been used in this work. Basing on the spectral line obtained by OES and combining with the current literature, OH, O and four carbonaceous species

(C, CN, CH and C₂) may be formed or react with other species through many reaction processes which is especially complicated at atmospheric pressure. However, we cannot get the exact atomic and molecular process in Ar/C₂H₅OH APPJ with current experimental and theoretical condition. Some main excited species were identified and summarized in table 1 for the plasma discharge process of Ar/C₂H₅OH.

In Ar/C₂H₅OH plasma, four types of chemical bonds contained in ethanol molecules can be broken by electron impact, and lead to generation of C₂H₅, H, OH, CH₂OH, CH₃ and three isomeric radicals of C₂H₅O (CH₃CHOH, CH₂CH₂OH and CH₃CH₂O), etc through the following reactions with comparable probabilities [28, 29]:



The ionization of C₂H₅OH has been ignored because of low degree of ionization [28, 30]:

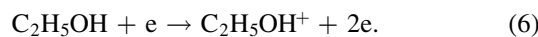


Table 1. Excited species identified in Ar/C₂H₅OH plasma.

Species	Transition	Position (nm)	References
C		247.8	[29]
CN	$B^2\Sigma \rightarrow X^2\Sigma$	386.2, 387.1, 388.3	[30]
CH	$A^2\Delta \rightarrow X^2\Pi$	431.3	[31]
	$C^2\Pi^+ \rightarrow X^2\Pi$	314.3	[31]
C ₂	$A^3\Pi \rightarrow X^3\Pi$	450–600	[32]
OH	$A^2\Sigma^+ \rightarrow X^2\Pi$	308.9	[33]
H _α	$3d^2D \rightarrow 2p^2P^0$	656.3	[34]
H ₂	$d^3\Pi_{\mu} \rightarrow a^3\Sigma^+_g$	580–650	[35]

These species are the primary products of ethanol dissociation by electron impact. They will participate in a series of reaction processes, so these primary species are considered firstly. These radicals can be further impacted by electron, and dissociate into smaller free radicals through the reactions in [28]. They could also react with each other or react with ethanol molecules through the reactions in [28, 30–37].

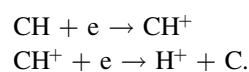
The C₂ Swan bands are at 469.7, 471.5, 473.7, 512.9, 516.4, 550.2, 554.0, 558.5 and 563.5 nm. In figure 3, the high resolution spectra of four carbonaceous species, namely, the C band at 247.8 nm, the CN band at 388.3 nm, the CH band at 431.3 nm and the C₂ Swan band at 516.4 nm, are presented. The emission lines of CH and C₂ have also been observed in plasmas for Ar/CH₄ and Ar/C₂H₂ mixtures, but C and CN have not been observed [38]. Through examination of these emission lines measured under different condition, we find that those high resolution spectra are independent of the ethanol content and the RF power. This result shows that the rotational distribution of excited levels of four carbonaceous species did not change with the ethanol content and the RF power. It is in good agreement with the results in literature [38]. Because this plasma discharge is operated in open air, CN will be generated by the reactions of carbonaceous radicals with N₂ and dissociated N from ambient air.

The important information about chemical and physical processes that occurring in the plasma was discussed by the emitted spectral lines. However, a drawback of OES is that nonemissive species cannot be observed. An increase of ethanol content results in a decrease of all Ar emission lines (figure 2). This can be correlated with a decrease in electron temperature (T_e) at higher ethanol content. Figure 4 shows the optical emission intensities of six different species as a function of the concentration of ethanol. In figure 4(a), an increase in ethanol content resulted in a decrease of Ar (772.3 nm) emission intensity. As well known, the effect of the presence of molecular gases in noble gas plasma. The addition of molecular gases into the noble gas discharge plasma will result in a change of energy efficiency which transfers between electrons and heavy species that causes a reduction in T_e [39]. The electron energy required to dissociate the C₂H₅OH molecule is 7–8 eV and the energy of the radicals dissociation is lower than the energy of Ar excitation [40]. The radicals in plasma can also cause a

decrease in T_e . So the radicals also have quenching effect for the ethanol plasma discharge. The energy of Ar excitation is about 13.08–13.33 eV (related with the transition) and its ionization energy is larger than 14 eV and ethanol is 10.5 eV. Hence, the T_e decreased with the ethanol content increase will affect intensively the intensities of Ar. The excited N₂ observed from emission lines are all N₂ $C^3\Pi_{\mu} - B^3\Pi_g$ ($v_1 = 0, 1, 2,$ and 3 and $v_2 = 0, 1, 2,$ and 3) transitions. The excitation energy of the N₂ $C^3\Pi_{\mu}$ states for $v_1 = 0$ to 3 are 11.178 eV, 11.425 eV, 11.668 eV and 11.908 eV, respectively, and the electron energy which is required to produce O from H₂O is 15.9 eV. Those energy are larger than the energy of the ethanol molecule dissociation, so the quenching effect of ethanol molecules also has a strong effect on the emission intensities of N₂ and O. The emission of N₂ and O lines will not be observed when ethanol content is larger than 1500 ppm. The effect of the molecular gases mixed in noble gas plasma has also been observed when other molecular species are added [38, 41, 42]. The emission intensity of OH (308.9 nm) has a nonlinear relation with ethanol content, and the maximum value at about 1100 ppm. In a pure Ar plasma, OH emission seen in figure 2 is coming through reaction with water in the air [39]. The density of OH can be increased when adding some ethanol. This is due to that there are other channels to produce OH. However, the increase of ethanol content would lead to a decrease of T_e .

In figure 4(b), when ethanol content is larger than 1000 ppm, the emission intensity of CH keeps unchanged with the ethanol content which means that the CH reaches a saturated condition. The increase of C, C₂ and OH is due to the dissociation of ethanol. The increase of CN means that the reactions which can produce CN radicals are promoted by the increase of ethanol content. On the other hand, the energy of dissociation of the ethanol molecule is lower than that of the excitation of Ar, adding ethanol steam can result in a loss of the electrons. With the ethanol content increase, there have not enough electrons to maintain the primary plasma discharge. So the high ethanol content makes the decrease of emission intensities of C, CN, C₂ and OH when the ethanol content is larger than 3700 ppm. The mixture plasma discharge with RF input power of 70 W, will be quenched when ethanol content is higher than 4600 ppm.

Figure 5 illustrates the variation of C (247.8 nm)/C₂ (516.4 nm), CN (388.3 nm)/C₂ (516.4 nm) and CH (431.3 nm)/C₂ (516.4 nm) emission intensity ratios as a function of the ethanol content from 0 to 4600 ppm. These ratios are much lower than that in [42]. These three curves have the similar variation tendency, gradually decreasing with the ethanol content increase, which indicated that the higher ethanol content is inclined to create the C₂ radicals. The CH radicals can be broken and dissociated by electron impact and form free radicals. These free radicals and fraction can react with other radicals and produce C or CN species. The reaction is following:



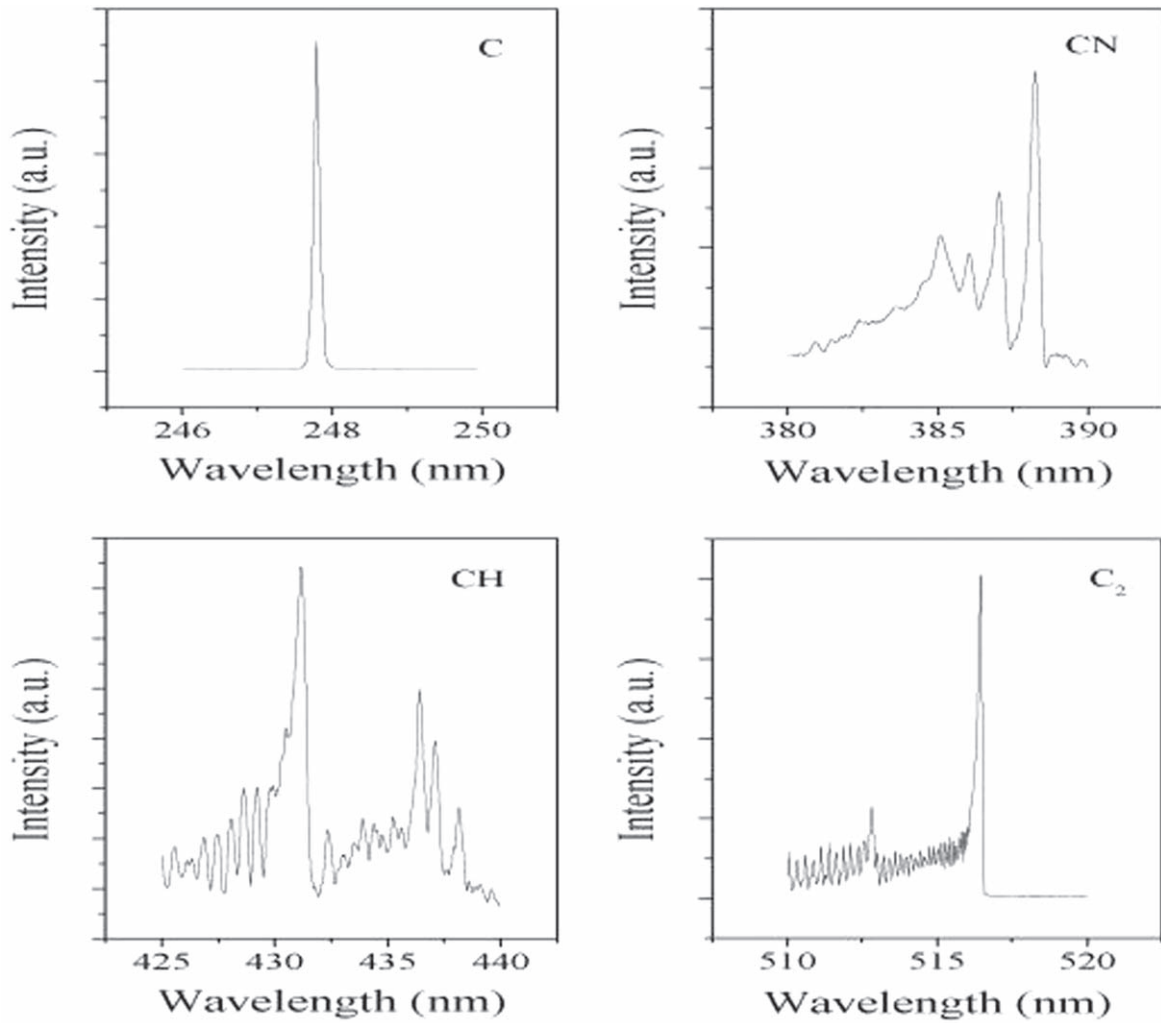


Figure 3. High resolution spectra of four carbonaceous species: C, CN, CH and C₂ for identical concentration of ethanol (3118 ppm) at 70 W.

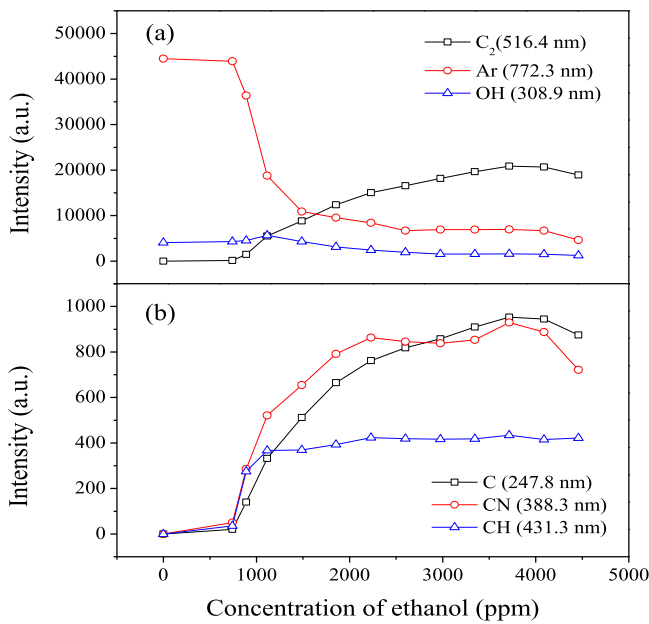


Figure 4. Optical emission intensity of excited species as a function of the concentration of ethanol at RF power of 70 W.

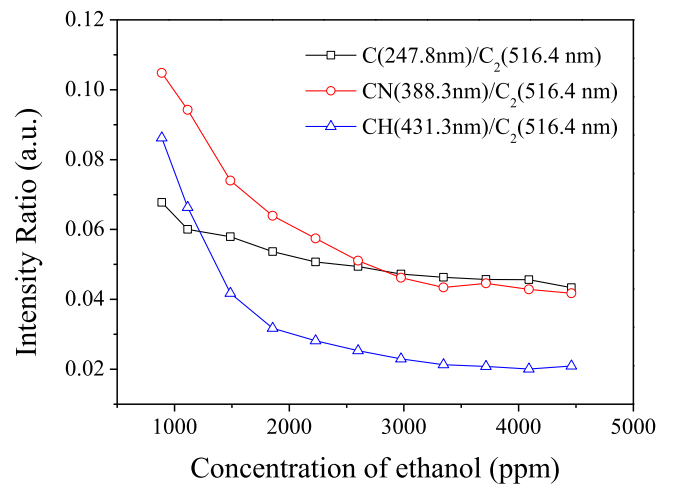


Figure 5. Intensity ratios in plasma as a function of the concentration of ethanol at RF power of 70 W.

C species can be combined into C₂. As a consequence, it is inclined to create the C₂ compounds with the increase of ethanol content. The emission intensity ratio of C₂ (516.4 nm)

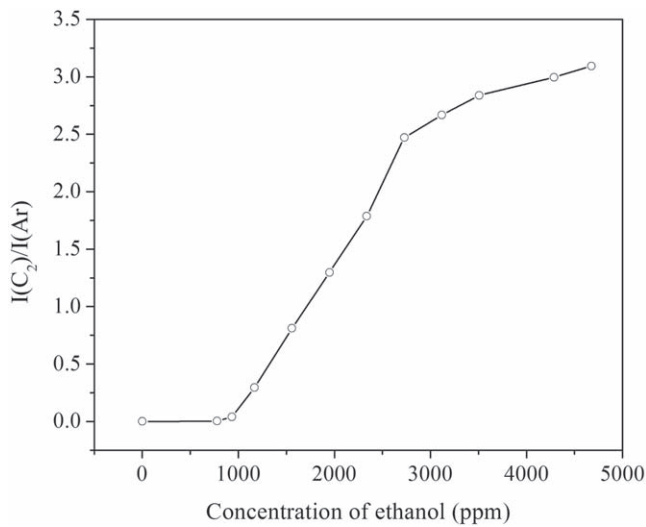


Figure 6. Intensity ratio of C₂ (516.4 nm) with respect to the Ar (772.3 nm) in plasma as a function of the concentration of ethanol at RF power of 70 W.

with respect to Ar (772.3 nm, 2P₂-1S₃) increase with the increase of ethanol content, as shown in figure 6. The 772.3 nm is one of the strong lines of Ar, so it is selected to be compared with. The C/Ar, CN/Ar and CH/Ar emission intensity ratios have similar variation tendency with the emission intensity ratio of C₂/Ar. This indicates that although the content of ethanol is less than Ar, ethanol molecules and their fragments are easily dissociated by electron. The electron energy decays as the ethanol content increases due to the electron impact processes to ethanol molecules and their fragments. In [42], we had studied the OES of the same carbonaceous radicals with the similar hydrocarbon (methanol, CH₃OH) and obtained the similar results. However, the RF excitation frequency (33 MHz) in this paper is much higher than that in [42] (2 MHz). From this research and the [42], it seems that the driving frequencies in the range of 2–33 MHz have little influence on the carbonaceous radicals. Yet, it still needs more experimental research to confirm. The RF excitation frequencies which are higher than 13.56 MHz had rarely been used in atmospheric pressure plasma. Furthermore, the effect of RF power on the radicals was studied and these methods will be used to deposit film in research.

Controlling the RF glow discharge in the α mode is very important to obtain homogeneous and low temperature plasma [43]. The RF plasma jet device can maintain stable α mode discharge in the range of less than 110 W, and it will tend to the α-γ mode transition with the further increase in the RF power. Figure 7 showed that the emission intensities of C₂ are increasing with the variation of the RF input power, which is in stable α mode discharge of the three different ethanol contents. The emission intensities of C, CN, and CH also are increasing (not shown here). This means that the high RF power is favor of generating the excited species. In figure 8, however, the ratios of C/C₂, CN/C₂ and CH/C₂ emission do not change significantly with the increase of RF input power for stable discharge mode.

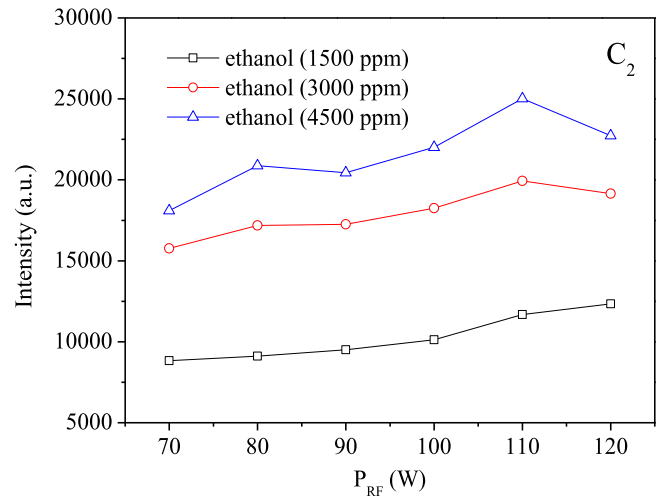


Figure 7. Emission intensity of C₂ as a function of the RF power at the total gas flow of 4 slm.

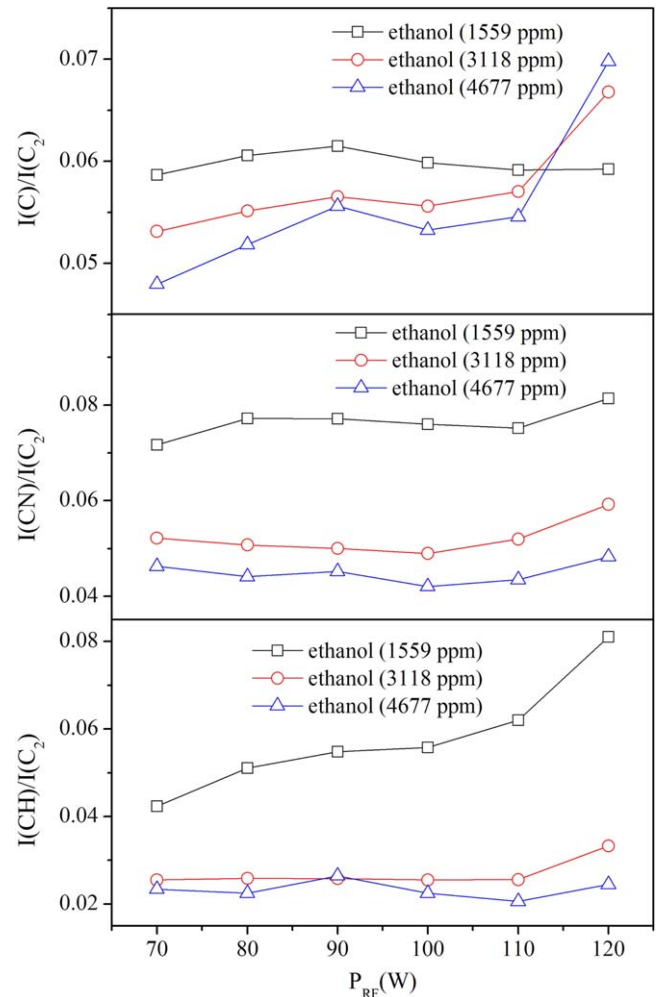


Figure 8. Intensity ratios versus the change of RF power at the total gas flow of 4 slm.

When it was ignited for 10 min, a layer of black films was deposited on silicon wafer with 1 mm distance of source-substrate. Finally as a supplement, the scanning electron microscopy (SEM) and x-ray photoelectron spectroscopy

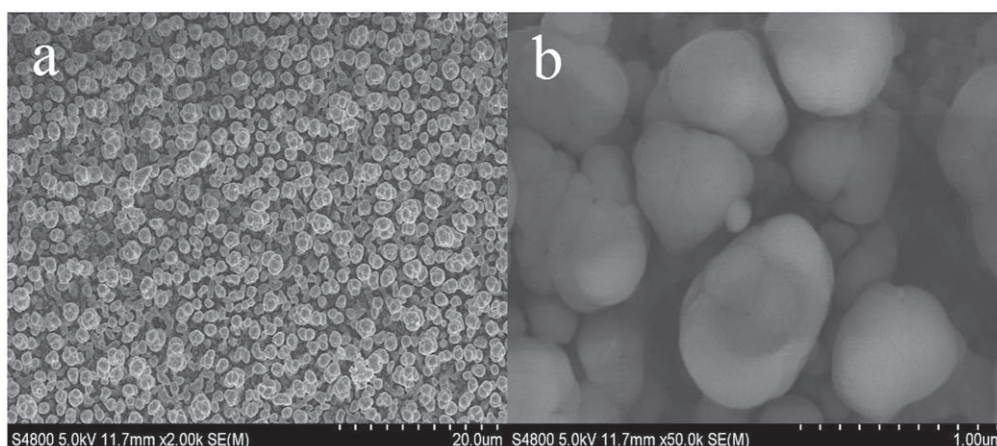


Figure 9. SEM image on the deposit surface: (a) low and (b) high magnifications.

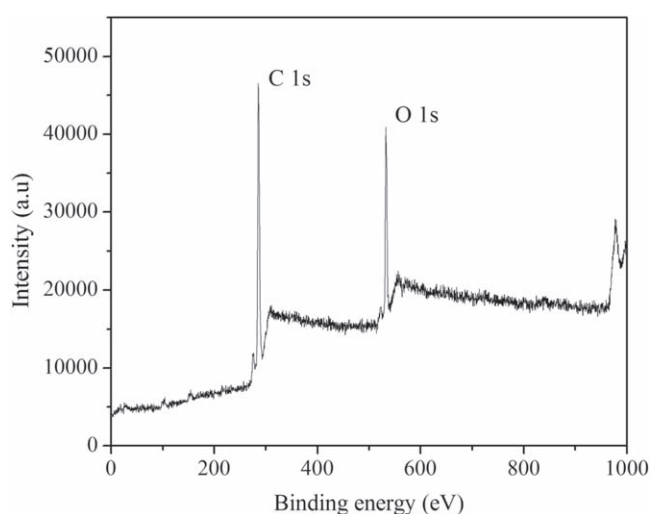


Figure 10. XPS survey scan of deposits.

(XPS) of deposition films were given in figures 9 and 10 respectively. The deposition time was 600 s. The ethanol content of the plasma jet was 3700 ppm. The RF input power was 70 W. Figures 9(a) and (b) show low and high magnification images obtained with the SEM (FESEM, Hitachi S-4800). It can be seen that the spherical particles are agglomerated into clusters. The diameters of the clusters are about several hundred nanometers. Figure 10 shows the XPS (PHI-5702) survey scan of the sample. This spectrum clearly indicated the C and O were presented in these films. The existence of oxygen in the sample maybe comes from OH or environmental O, or Si–O–Si bonds due to the oxidation of the silicon substrate. In order to prepare different carbonaceous films by using ethanol, further studies will concern to control the content of a specific carbonaceous species (C, CN or CH) through adding other gas, such as nitrogen, hydrogen.

4. Conclusion

In summary, we investigated the excited species of Ar/C₂H₅OH plasma at atmospheric pressure by OES. The

results show that introduction of ethanol leads to generation of four excited carbonaceous species in Ar/C₂H₅OH plasma, namely, C, CN, CH and C₂. The concentration of ethanol increased which is beneficial to increase emission intensities of four carbonaceous species. However, the higher ethanol content is, the more easily, it is inclined to create more C₂ radicals. The emission intensity ratios of C/C₂, CN/C₂ and CH/C₂ have no significant change with the RF input power increase.

Acknowledgments

The authors gratefully acknowledge the support provided by National Natural Science Foundation of China (No. 11165012), Project of Natural Science Foundation of GanSu province (No. 145RJZA159), China Postdoctoral Science Foundation funded project (Nos. 2011M501494 and 2012T50831).

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