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Investigation on the characteristics of an atmospheric-pressure microplasma plume confined inside a long capillary tube

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Abstract

An atmospheric-pressure microplasma plume of diameter 10 μm is generated inside a long tube. The length of the microplasma plume reaches as much as 2 cm. First, with the assistance of an air dielectric barrier discharge (DBD), the ignition voltage of the microplasma decreases from 40 kV to 23.6 kV. Second, although the current density reaches as high as \((1.2 - 7.6) \times 10^4\) A m\(^{-2}\), comparable to the current density in transient spark discharge, the microplasma plume is non-thermal. Third, it is interesting to observe that the amplitude of the discharge current in a positive cycle of applied voltage is much lower than that in a negative cycle of applied voltage. Fourth, the electron density measured by the Stark broadening of Ar spectral line 696.5 nm reaches as high as \(3 \times 10^{16}\) cm\(^{-3}\), which yields a conductivity of the microplasma column of around 48 S m\(^{-1}\). In addition, the propagation velocity of the microplasma plume, obtained from light signals at different axial positions, ranges from 1 \(\times 10^5\) m s\(^{-1}\) to 5 \(\times 10^5\) m s\(^{-1}\). A detailed analysis reveals that the surface charges deposited on the inner wall exert significant influence on the discharge behavior of the microplasma.

Keywords: microplasma, atmospheric pressure plasma, non-thermal plasma, microdischarge

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

1. Introduction

Atmospheric-pressure microplasma jets are receiving considerable attention due to their advanced potential applications, such as in plasma medicine [1–3], nanofabrication [4], and material processing [5–8]. By using AC or pulsed high voltage with a repetition frequency of several kHz, helium or argon microplasma jets are usually generated inside a dielectric tube and then propagate outward into the open air. The length of a plasma plume in the open air can be as much as 10 cm [9]. This allows the remote delivery of active species (electrons, oxygen-related species and nitrogen-related species) to the local surface of the sample, without limitation of the size of the sample.

From the view of application, the smallest possible diameter of the microplasma jet is very much desired in some circumstances, such as single-cell-precision cancer treatment [10] or micro-patterning a functionalized surface [5]. One effective approach to minimize the plasma jet is to reduce the diameter of the dielectric tube. By using a cone nozzle of small diameter, several groups have developed microplasma jets with diameters of 1–50 μm for various applications [11–13]. However, little attention has been paid to the gas discharge physics when the diameter of microplasma jets is reduced.

To better understand the effects of the tube diameter on the characteristics of microplasma jets, great efforts were made in several previous works [14–20]. In 2013, Ji et al studied the characteristics of a microplasma plume confined inside a glass tube [14]. With the reduction of the tube diameter from 2000 μm to 100 μm, the current density grew rapidly. In 2014, Jogi et al reported that when the diameter of
a microplasma jet decreases from 500 μm to 80 μm, the ignition voltage, vibrational temperature and electron density increase, while the gas temperature is almost unchanged [15]. They believed that the surface charges on the wall and the diffusion of long lifetime active species significantly affect the discharge behavior. By 2D numerical simulations, Cheng et al. found that the increasing ignition voltage with the reduction of tube diameter is caused by the low density of seed electrons [16]. As the tube diameter decreases to 600 μm, the electric field increases up to 20 kV cm⁻¹, and the propagation of the helium microplasma converts from a ‘plasma bullet’ to a continuous plasma column, as reported by Wu et al [17]. Most of these works focus on tube diameters in the range of 80–2000 μm. For tube diameters of less than 80 μm, there is a lack of investigation into the characteristics of microplasma jets. Recently, we developed an Ar microplasma plume confined inside a small tube with diameter of 6 μm [18]. The electron density is of the order of 10¹⁶ cm⁻³, which is 2–4 orders of magnitude higher than those with diameters of several mm. The peak-to-peak value of AC ignition voltage is as high as 40 kV. Gou et al. studied a helium microplasma plume generated in a micro quartz tube, with diameters varying from 245 μm to 6 μm [19]. The electron density reached as high as 10¹⁵–10¹⁶ cm⁻³. They predicted that an electron density as high as 10¹⁸ cm⁻³ will be obtained if the tube diameter is further reduced to 1 μm. They also believed that the plasma sheath between the bulk plasma and the wall exerts significant influence over the discharge behavior. In our previous work, an array of microplasmas plumes was demonstrated by using a photonic crystal fiber [20]. The diameter of each microplasma column was as small as 3.4 μm. The electron density estimated by the Ar Stark broadening method was up to 8 × 10¹⁶ cm⁻³. It was indicated that the Debye shielding effect might not be applicable in a microplasma array of such small diameter. This is consistent with Eden’s prediction of a breakdown of quasi-neutrality when the plasma size is close to 1 μm [21]. Therefore, it would be interesting to understand the underlying discharge physics of the microplasma plume for fundamental atmospheric-pressure plasma phenomena.

In this work, an atmospheric-pressure microplasma plume of diameter 10 μm is generated inside a capillary tube. By using an air dielectric barrier discharge (DBD)-assisted discharge, the peak-to-peak value of AC ignition voltage is reduced to 23 kV, which is much lower than those in previous work [18–20]. The electron density estimated from both the Ar Stark broadening method and the current density is at a level of 10¹⁶ cm⁻³. The propagation velocity of the microplasma plume is (1–5) × 10³ m s⁻¹. A detailed discussion of the propagation mechanism of the microplasma plume is presented.

2. Experimental setup

As shown in figure 1, a stainless steel needle serves as the high voltage electrode, being inserted into a quartz tube with a length of 5 cm. The diameter of the needle is 0.25 mm, which is smaller than the 0.35 mm inner diameter of the quartz tube. The needle is directly connected to the output of an AC power supply (CTP-2000 K). The amplitude of the applied voltage reaches up to 30 kV. The frequency of the AC voltage is fixed at 2 kHz. A capillary tube of inner diameter 10 μm and outer diameter 80 μm is inserted into the quartz tube. The length of the capillary tube is as much as 8 cm. The distance between the left end of the capillary tube and the right end of the quartz tube is set at 1 mm. The connection between them is sealed to prevent gas leakage. The distance between the needle tip and the left end of the capillary tube is about 1 mm. To assist the microdischarge inside the quartz tube, air DBD is generated just above the quartz tube and wrapped around it. A grounded ring electrode, made of copper foil with a width of 4 mm, is attached to the glass tube. The horizontal distance between the copper foil and the right end of the quartz tube is 6 mm. The gap distance of the DBD is fixed at 1 mm. The DBD provides UV radiation and strengthens the external electric field, which reduces the ignition voltage from 40 kV (without DBD) to 23 kV.

The working gas is argon. The gas flow is controlled by a pressure barometer (YB-150) located at a position close to the gas inlet of the quartz tube. The gas pressure is fixed at 0.008 MPa above atmospheric pressure. According to Poiseuille’s law, the pressure drop \( \Delta p \) inside the capillary tube is given by [22]:

\[
\Delta p = \frac{128 \eta Q}{\pi D^4} \Delta l
\]

where \( D \) is the inner diameter of the capillary tube, \( Q \) is the flow rate, \( \Delta l \) is the length of the capillary tube, and \( \eta \) is the viscosity coefficient. For \( D = 10 \) μm, \( \Delta l = 8 \) cm, \( \Delta p = 0.008 \) MPa, and \( \eta = 22.3 \mu Pa s \) at room temperature, the flow rate is calculated as \( 6.6 \times 10^{-5} \) ml min⁻¹. Therefore, the Reynolds number can be evaluated as 0.01. It means that the gas flow inside the capillary tube is laminar.

The output voltage of the AC power supply is measured by a HV probe (Tektronix P6015A). To monitor the discharge current of DBD, current probe 2 (Pearson 2877) and a
matching non-inductive resistor \((r)\) with a value of 930 \(\Omega\) are used. For measurements of the discharge current carried by the microplasma plume, current probe 1 (Pearson 6585) is applied. The axial position of current probe 1 is 10 mm from the right end of the quartz tube. The current and voltage waveforms are recorded by a Tektronix MDO3034 digital oscilloscope having a sampling rate of 2.5 GS s\(^{-1}\).

The optical emission spectrum of the air discharge is measured by a spectrometer (Andor Technology Spectrograph SR500i) with an intensified charge-coupled camera detector (ICCD, iStar 334T). The exposure time of the ICCD is 0.5 s. The gated width and the gain are 50 \(\mu\)s and 2500, respectively. An optical lens \((f = 50 \text{ mm})\) is mounted between the air plasma source and the entrance slit of the spectrometer, resulting in an almost 1:1 projection of the plasma onto the entrance slit. The slit width and the grating are fixed at 50 \(\mu\)m and 1200 \(g \text{ mm}^{-1}\), respectively. To observe the temporal behavior of microplasma propagation, a photomultiplier tube (Hamamatsu H10721-01) is used to detect the light emitted from the microplasma. The photomultiplier tube (PMT) is connected to an optical fiber having a hole of diameter 0.2 mm. By moving the position of the PMT fiber, the emitted light signals from different positions of the microplasma plume are obtained. Therefore, knowing the time delay between these light signals, the propagation velocity of the tip of the microplasma plume can be determined. Because the plasma radiation is synchronized naturally with the discharge current, the current waveform serves as the time reference signal for the determination of the time delay between these PMT signals.

3. Experimental results

3.1. Effects of applied voltages

When Ar gas is fed into the capillary tube and a high voltage is applied, a microplasma plume will be generated inside the capillary tube. In figure 2, there is no microplasma plume inside the capillary tube if the applied voltage is lower than 22 kV. When the applied voltage is increased to 23.6 kV, a microplasma plume is ignited. This ignition voltage is much lower than the 40 kV reported in references \([18–20]\). As the voltage increases from 23.6 kV to 30 kV, the length of the microplasma plume increases from 1.5 mm to 20 mm, as shown in figure 3. The length of the microplasma plume is determined by the faint glow in the plume tip, which causes the uncertainty in the plume length. An error bar is therefore used in figure 3. For the tube diameter of 10 \(\mu\)m, the aspect ratio (length/diameter) of the microplasma plume reaches up to 2000, which is higher than those reported by Ji et al \([14]\) and Jogi et al \([15]\). In addition, it should be pointed out that when the applied voltage is as high as 26.8 kV, the air plasma around the capillary tube is excited along with the generation of the microplasma plume. The ignition location \((x)\) of the air plasma is about 1.5 mm away from the right end of the quartz tube. It is indicated that the electric field strength is extremely high at \(x = 1.5 \text{ mm}\), instead of \(x = 0 \text{ mm}\).

![Figure 2. Photographs of microplasma plumes at different applied voltages. The value of the applied voltage in each image is peak-to-peak. All photos are taken by a digital camera (Nikon D7100) with an exposure time of 2 s.](image)

![Figure 3. The length of the microplasma plume \((L_p)\) as a function of the applied voltage.](image)

3.2. Electrical characteristics

Figure 4 shows the \(I–V\) waveforms. For the current \((I_p)\) carried by the microplasma plume, one or two current pulses per half-cycle of applied voltage are observed. The onset time with respect to the voltage of zero and the amplitude of discharge current is not constant, which indicates the randomness of the microdischarge inside the capillary. The amplitude of \(I_p\) is in the range of 10–60 mA, which yields current densities in the range of \((1.2–7.6) \times 10^4 \text{ A cm}^{-2}\). This is close to the current density in transient spark discharge \([23]\). It is interesting to find that the amplitude of the discharge
current in the negative cycle is much higher than that in the positive cycle. On the other hand, for the current \( I_g \) of air DBD, multiple current pulses per half-cycle of applied voltage are observed. When the current pulse of \( I_g \) occurs, the current pulse of \( I_p \) will appear. Figure 5 shows the single current pulses of \( I_p \) and \( I_g \). The two current pulses appear at almost the same time. The pulse widths of \( I_p \) and \( I_g \) are almost the same. The pulse width of \( I_p \) for a positive discharge is 115 ns, which is twice times larger than that in a negative discharge. By integrating a single current pulse with time, the charges deposited into the microplasma plume are estimated at 2.6 nC. In addition, the dissipated powers to sustain the microplasma plume and DBD are obtained from the current integrating method. The dissipated powers are calculated from integrating the multiple discharge current pulses in four voltage cycles, in which each current pulse is integrated separately to exclude the effects of displacement current. The dissipated power is calculated to be 30.5 mW for the microplasma plume and 57.5 mW for DBD. To confirm this estimate, a Q-V Lissajous method is adopted. The charges are determined from the voltage drop on a film capacitor, which is connected in series between air DBD and matching resistor. The capacitance is 1 nF. As shown in figure 6, the dissipated power on air DBD is 105 mW, which is greater than the power estimated by the current integrating method.

3.3. Propagation of the microplasma plume

To capture the dynamics of the microplasma plume, a PMT is used to detect the plasma radiation. Figure 7 shows the PMT signals at different axial positions, which are synchronized with the discharge current. The negative current pulses of PMT signals are clearly identified. The smaller the axial position is, the earlier the PMT signal appears. Therefore, the time delay between these PMT signals at different positions is obtained. The details of the propagation velocity of the microplasma plume as a function of axial position are plotted in figure 8. Due to the instability of the discharge, the initiating time of the PMT signal, corresponding to the peak of the PMT pulse signal, jitters within a certain range for each current pulse. By carrying out the same experiment four times, the uncertainty of the initiating time of the PMT signal is estimated to be less than 4 ns. Thus, the error bar is estimated by carrying out the same experiment four times. As the tip of the microplasma plume propagates from \( x = 3 \) mm to \( x = 17 \) mm, the velocity increases first and then decreases after reaching the maximum of \( 5 \times 10^5 \) m s\(^{-1} \) at \( x = 9 \) mm. Therefore, the propagation velocity is \((1-5) \times 10^5 \) m s\(^{-1} \), which indicates that the microdischarge inside the capillary tube behaves like a streamer discharge, instead of a Townsend discharge.

3.4. Electron density

Figure 9 shows the emission spectra of the Ar microplasma plume confined inside the capillary tube of diameter 10 \( \mu \)m. It is clearly shown that the emission spectra of the microplasma are dominated by the Ar excited states, such as Ar\( (2p1) \), Ar\( (2p2) \), Ar\( (2p3) \), Ar\( (2p6) \), Ar\( (2p8) \), and Ar\( (2p9) \). The emission intensity of N\( _2 \) excited states is negligible with respect to the Ar excited states. It is indicated that the effects of N\( _2 \) impurity in Ar gas on the plasma properties can be ignored. On the other hand, the gas temperature can be estimated by fitting the emission spectra of N\( _2 \) second positive system 0–0 transition. It reveals that the gas temperature of the microplasma plume is around 350–500 K in our previous work [18–20]. By using a collisional-radiative model for Ar excited levels, the electron temperature of the microplasma plume can be determined. A detailed description of this method can be found in reference [24]. Briefly, this model is based on the assumption that the population of four of the 4p levels of Ar is dominated by electron excitation kinetics, and the main process of deexcitation is spontaneous radiative decay. In this work, the 2p2, 2p6, 2p8, and 2p9 levels of Ar excited states are chosen. The transition probabilities and cross-section for electron collision excitation from the ground state and metastable levels can be found in references [25, 26]. The electron temperature is estimated to be 1.6 eV.

With knowledge of the electron temperature, the electron density of the microplasma can be determined by Stark broadening of Ar spectral line 696.5 nm [27]. In addition to Stark broadening, Doppler broadening, instrumental broadening, van der Waals broadening, resonance broadening and natural broadening also contribute to the broadening of the Ar spectral line profile. In atmospheric-pressure microplasmas, the resonance broadening and the natural broadening are negligible. The spectral line profiles caused by these broadening mechanisms can be well approximated by Lorentzian forms (van der Waals broadening and Stark broadening) and Gaussian forms (Doppler broadening and instrumental broadening). A Voigt profile is obtained from a convolution of the Gaussian and Lorentzian forms. Therefore, by fitting the experimental curve with a Voigt profile, the Stark broadening width can be determined with the knowledge of other broadening widths. Detailed procedures of this method can be found in our previous work [20]. The instrumental broadening width estimated by a low-pressure Hg lamp is 0.087 nm. The Doppler broadening is induced by the thermal motion of the emitters [27]. The width can be estimated to be
1.7 × 10⁻³ nm for a gas temperature of 500 K, which is negligible compared to the instrumental broadening width. The van der Waals broadening comes from a dipolar interaction between the emitter and the neutral perturbers [27], the width of which is calculated as 0.026 nm for a gas temperature of 500 K. The relationship between the Stark broadening

\[
\Delta \lambda_T = 2w(T_e)n_e[1 + 1.75 \times 10^{-4}n_e^{0.25}\alpha(T_e)\times(1 - 0.068n_e^{0.16}T_e^{-0.5})] \times 10^{-16}
\]

where \(w(T_e)\) is the electron impact half-width and \(\alpha(T_e)\) is the static ion-broadening parameter. As shown in figure 10, the Stark broadening width of Ar spectral line 696.5 nm is 0.039 nm. Therefore, the electron density of the microplasma plume is calculated to be 3 × 10¹⁶ cm⁻³ for the electron temperature of 1.6 eV. This is 2–4 orders of magnitude higher than those in mm-sized plasma jets [28–30].
An atmospheric-pressure microplasma plume of diameter 10 μm and length 2 cm is generated inside a long tube. With the assistance of air DBD, the ignition voltage of the microplasma is reduced to 23.6 kV, which is much lower than the 40 kV reported in our previous work [18–20]. This is probably due to the growth of the electric field with the additional ring electrode. Because of seed electrons provided by the photo-ionization, the UV radiation from air DBD may also lower the ignition voltage, similar to the results in a helium corona-assisted air discharge [31]. The current density reaches as high as (1.2–7.6) × 10^4 A cm⁻², which is comparable to the current density in transient spark discharge [23]. However, the gas temperature is much lower than that in spark discharge. This is probably due to the large surface-to-volume ratio and the wall thickness as small as 80 μm, so that the heat transfer between the microplasma and ambient air is rapid through the tube wall. Thus, the heat loss on the wall may be important for sustaining the non-equilibrium nature of the microplasma plume, rather than the transition to hot spark discharge. Moreover, it is interesting to observe that the amplitude of the discharge current in the negative cycle of applied voltage is much higher than that in the positive cycle of applied voltage, while the pulse width of the discharge current is just the opposite. The temporal behavior of the microplasma plume shows that the propagation velocity is in the range of (1–5) × 10^3 m s⁻¹, which is similar to the streamer discharge [23]. The electron density measured by the Stark broadening of the Ar spectral line reaches as high as 3 × 10^16 cm⁻³, which is 2–4 orders of magnitude higher than those in mm-sized plasma jets [28–30].

To confirm the high electron density of the microplasma plume, a calculation of electron density based on current density is carried out. The relationship between the current density (j) and the electron density in weakly ionized plasmas is given by [23]:

\[ j = \varepsilon n_e v_d \]

where \( v_d \) is the electron drift velocity. Because the propagation of the microplasma plume is governed by the movement of electrons under a high electric field in front of the plume tip, the electron drift velocity is comparable to the propagation velocity of the microplasma plume. Therefore, the \( v_d \) can be roughly equal to the propagation velocity. For a current density of (1.2–7.6) × 10^4 A cm⁻², propagation velocity of (1–5) × 10^3 m s⁻¹ and \( \varepsilon \) of 1.6 × 10⁻¹⁹ C, \( n_e \) is estimated to be (0.15–2.4) × 10¹⁶ cm⁻³. This value is somewhat lower than the electron density obtained from the Stark broadening method. The underestimate may come from the randomness of the microdischarge inside the tube, without consideration of the charge diffusion to the wall, and the rough determination of the electron drift velocity. In addition, the electric field estimated from the propagation velocity of 3 × 10^3 m s⁻¹ is about 100 kV cm⁻¹, which is high enough to break down ambient air [23]. That is exactly observed in figures 2(d) and (e).

The conductivity of the microplasma plume is described by Ohm’s law [23]:

\[ \sigma = \frac{n_e e^2}{m_e \varepsilon c} = \varepsilon n_e \mu_e \]

where \( m_e \) is the electron mass and \( \varepsilon_c \) is the collision frequency between electrons and neutral particles. For an electron mobility of 3 × 10⁷ cm²/(Vs) and an electron density of 10¹⁶ cm⁻³ in the microplasma plume, the conductivity is estimated to be 48 S m⁻¹. The resistance for a microplasma column of length 1 cm is 2.65 MΩ. It is indicated that the microplasma plume is not a perfect conductor. Therefore, most of the applied voltage drops on the microplasma column behind the plume tip as the microplasma plume propagates forward. This limits how far the microplasma plume reaches. To achieve a longer microplasma plume, the applied voltage has to be higher, which is consistent with the results in figure 2. In addition, to minimize the resistance, the electron density should be higher.

To understand the propagation behavior of the microplasma plume, the electric field in front of the tip of the microplasma plume is studied. According to the analysis of conductivity of microplasma, the plasma channel left behind the plume tip is not a perfect conductor. Therefore, to simulate the electrostatic field distribution in front of the plume tip, two extreme cases are considered. One case assumes the microplasma column to be a perfect conductor, without consideration of the local electric field induced by charges. The applied voltage on the conductor is 15 kV. The other case replaces the conductor with charges, without an applied voltage. The density of the charges is 3 × 10¹⁶ cm⁻³. By using COMSOL Multiphysics 5.0 commercial software [32], the electrostatic field distributions for both cases are shown in figure 11. It is clearly shown that the local electric field induced by the charges in figure 11(b) is higher than the external electric field in figure 11(a). Both electric fields in front of the head of the conductor or the charges reach a level of 10⁷ V m⁻¹, which yields an electron drift velocity of 3 × 10⁻⁶ m s⁻¹. This is one order of magnitude higher than the propagation velocity in figure 8. This overestimate is possible when not considering surface charges deposited on the inner wall of the microtube.
where \( D_i \) is the Ar ion diffusion coefficient, \( T_e \) and \( T_i \) are 1.6 eV and around 500 K, respectively. Therefore, \( D_a \) is estimated to be 2.56 cm\(^2\) s\(^{-1}\). The mean electron lifetime with respect to the diffusion loss is \( \tau_3 = \frac{\Lambda}{D_a} \), where \( \Lambda \) is the characteristic diffusion length, which is given by [23]:

\[
\left( \frac{1}{\Lambda} \right)^2 = r^2 + \left( \frac{2.405}{R} \right)^2
\]

where \( L \) and \( R \) are the length and the radius of the microtube. For \( L \) of 8 cm and \( R \) of 5 \( \mu \)m, \( \tau_3 \) is estimated to be 16.9 ns. In other words, the electrons or ions in the microplasma diffuse to the wall very rapidly, and there charges accumulate or are lost through recombination. After the pulse discharge, and due to the mass difference between electrons and ions, the electrons are likely absorbed by the needle and the positive Ar ions are probably left inside the microtube. These positive ions, deposited on the inner wall of the microtube, induce a local electric field, the direction of which is towards the needle. This induced electric field hinders the positive discharge and strengthens the negative discharge, leading to a higher discharge current and a lower breakdown voltage in the negative discharge with respect to the positive discharge. That is exactly observed in figures 4 and 5. Moreover, the electric field induced by these positive ions may weaken the electric field in front of the tip of the plasma plume in a positive discharge, resulting in a reduction in electron drift velocity. That is why the propagation velocity is much lower than the electron drift velocity obtained from figure 11. Therefore, the charges deposited on the surface of the wall are thought to play an important part in the ignition and propagation of the microplasma plume inside the microtube.

5. Conclusion

In summary, an atmospheric-pressure microplasma plume of diameter 10 \( \mu \)m is generated inside a long tube. The ignition voltage is reduced to 23.6 kV with the use of an additional DBD, which is much lower than those in previous works [18–20]. The electron density obtained from the Stark broadening of Ar spectral line 696.5 nm reaches as high as \( 3 \times 10^{16} \text{ cm}^{-3} \), which is 2–4 orders of magnitude higher than those in mm-sized plasma jets [28–30]. The conductivity of the microplasma column is estimated to be around 48 S m\(^{-1}\), indicating that the microplasma plume is not a perfect conductor.

Although the current density of the microplasma plume is comparable to the current density in transient spark discharge, the microplasma plume remains non-thermal. This is probably due to the large surface-to-volume ratio and the thin wall, such that the thermal convection between the microplasma and ambient air is fast. By using PMT signals to characterize the temporal behavior of the microplasma plume, the propagation velocity is estimated to be (1–5) \( \times 10^3 \text{ m s}^{-1}\). The surface charges inside the microtube are expected to play a significant role in the ignition and propagation of the microplasma plume.

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