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How to Ignite an Atmospheric Pressure Microwave Plasma Torch Without any Additional Igniters

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Abstract:	<p>This movie shows how an atmospheric pressure plasma torch can be ignited by microwave powers with no additional igniters. After ignition of the plasma a stable and continuous operation of the plasma is possible and the plasma torch can be used for many different applications. On the one hand the hot (3600 K gas temperature) plasma can be used for chemical processes and on the other hand the cold afterglow (temperatures down to almost room temperature) can be applied for surface processes. For example chemical syntheses are interesting volume processes. Here the microwave plasma torch can be used for the decomposition of waste gases which are harmful and contribute to the global warming but are needed as etching gases in growing industry sectors like the semiconductor branch. Another application is the dissociation of CO₂. Surplus electrical energy from renewable energy sources can be used to dissociate CO₂ to CO and O₂. The CO can be further processed to gaseous or liquid higher hydrocarbons thereby providing chemical storage of the energy, synthetic fuels or platform chemicals for the chemical industry. Applications of the afterglow of the plasma torch are for example the treatment of surfaces to increase the adhesion of lacquer, glue or paint or the sterilization or decontamination of different kind of surfaces. The movie will explain what has to be done to ignite the plasma solely by microwave power without any additional igniters like for example electric sparks. The microwave plasma torch is based on a combination of two resonators - a coaxial one which provides the ignition of the plasma and a cylindrical one which guarantees a</p>

	continuous and stable operation of the plasma after ignition. The plasma can be operated in a long microwave transparent tube for volume processes or shaped by orifices for surface treatment purposes.
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10th October 2014

Dear editor,

the presented work explains how an atmospheric pressure microwave plasma torch can be ignited without any additional igniters and provides continuous and stable plasma operation. Atmospheric pressure plasma sources come more and more important for industrial processes since they offer a variety of different applications. The presented microwave plasma torch offers the opportunity of electrodeless plasma, an ignition of the plasma solely by the supplied microwave as well as a stable and continuous plasma operation. To achieve the ignition of the plasma without any additional igniters as well as the stable and continuous plasma operation the used power supply and the plasma torch have to be characterized in detail and carefully adjusted to each other. This process can be described in a common publication but it is much more clear and descriptive if all the needed procedures to characterize the plasma torch and the power supply and how they are adjusted carefully to each other are presented in a movie and therefore are published as a video article in JoVE. Furthermore, the ignition of the plasma is a very fast process and can only be observed with a high speed camera. Thus the publication by JoVE offers the opportunity to also illuminate this aspect and show the slow motion picture of the plasma ignition.

The main part of the presented work was conducted by Martina Leins and was supervised by Uwe Schumacher. Sandra Gaiser assisted with the high speed camera recording. Andreas Schulz and Matthias Walker supported the work with many helpful advices and fruitful discussions. Thomas Hirth is head of the institute and therefore is involved in the presented work.

The preparation of the manuscript was assisted by Mathew Solomon.

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Lastly, I would like to thank the editors for considering my work for publication in JoVE.

Thank you in advance and kind regards

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TITLE:

How to Ignite an Atmospheric Pressure Microwave Plasma Torch Without any Additional Igniters

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KEYWORDS:

atmospheric pressure plasma, microwave plasma, plasma ignition, resonator structure, coaxial resonator, cylindrical resonator, plasma torch, stable plasma operation, continuous plasma operation, high speed camera

SHORT ABSTRACT:

This movie shows how an atmospheric plasma torch can be ignited by microwaves with no additional igniters and provides a stable and continuous plasma operation suitable for plenty of applications.

LONG ABSTRACT:

This movie shows how an atmospheric pressure plasma torch can be ignited by microwave powers with no additional igniters. After ignition of the plasma, a stable and continuous operation of the plasma is possible and the plasma torch can be used for many different applications. On one hand, the hot (3600 K gas temperature) plasma can be used for chemical processes and on the other hand the cold afterglow (temperatures down to almost room temperature) can be applied for surface processes. For example chemical syntheses are interesting volume processes. Here the microwave plasma torch can be used for the decomposition of waste gases which are harmful and contribute to the global warming but are needed as etching gases in growing industry sectors like the semiconductor branch. Another application is the dissociation of CO₂. Surplus electrical energy from renewable energy sources can be used to dissociate CO₂ to CO and O₂. The CO can be further processed to gaseous or liquid higher hydrocarbons thereby providing chemical storage of the energy, synthetic fuels or platform chemicals for the chemical industry. Applications of the afterglow of the plasma torch are the treatment of surfaces to increase the adhesion of lacquer, glue or paint, and the sterilization or decontamination of different kind of surfaces. The movie will explain how to ignite the plasma solely by microwave power without any additional igniters, e.g., electric sparks. The microwave plasma torch is based on a combination of two resonators – a coaxial one which provides the ignition of the plasma and a cylindrical one which guarantees a continuous and stable operation of the plasma after ignition. The plasma can be operated in a long microwave transparent tube for volume processes or shaped by orifices for surface treatment purposes.

INTRODUCTION:

Atmospheric pressure microwave plasma torches offer a variety of different applications. On one hand they can be used for chemical volume processes and on the other hand their afterglow plasma can be applied for the treatment of surfaces. As surface treatment processes the treatment to increase the adhesion of glue, paint or lacquer or the decontamination or sterilization of surfaces can be named. The hot and reactive plasma itself can be used for

volume processes like the decomposition of waste gases¹⁻⁷. These waste gases are harmful, contribute to the global warming and can hardly be degraded conventionally. However, they are needed in growing industrial sectors such as the semiconductor branch. Other applications are chemical synthesis like the dissociation of CO₂ to CO and O₂ or CH₄ to carbon and hydrogen^{8,9}. Surplus electrical energy from renewable energy sources can be used to dissociate CO₂ into CO and O₂. The CO can be processed further to higher hydrocarbons which can be used as synthetic fuels for transportation, as platform chemicals for the chemical industry or as chemical storage.

There are some microwave plasma torches but most of them have disadvantages: They only have very small plasma volumes, need additional igniters, need cooling of the plasma reactor or can only be operated in pulsed mode¹⁰⁻¹⁸. The microwave plasma torch presented in this movie offers an ignition of the plasma solely with the provided microwave power with no additional igniters as well as a stable and continuous operation without any cooling of the plasma reactor for a broad range of operation parameters and can be used for all of the above mentioned applications. The microwave plasma torch is based on a combination of two resonators: a coaxial one and a cylindrical one. The cylindrical resonator has a low quality and is operated in the well-known E₀₁₀-mode with the highest electrical field in its center. The coaxial resonator is located below the cylindrical resonator and consists of a movable metallic nozzle in combination with a tangential gas supply. The high quality of the coaxial resonator exhibits a very narrow but deep resonance curve. Due to the high quality of the coaxial resonator a high electrical field can be reached which is required for the ignition of the plasma. However, the high quality of the coaxial resonator is associated with a very narrow resonance curve and therefore the resonance frequency has to perfectly match the frequency of the supplied microwave. Since the resonance frequency shifts after ignition of the plasma due to the permittivity of the plasma, the microwave can no longer penetrate into the coaxial resonator. For the continuous operation of the plasma the cylindrical resonator with a low quality and a broad resonance curve is needed.

An additional axial gas supply via the metallic nozzle of the coaxial resonator is possible. The plasma is ignited and confined in a microwave-transparent tube, for example a quartz tube. The permittivity of the quartz tube also affects the resonance frequency. Since the quartz has a permittivity of > 1, the volume of the cylindrical resonator is virtually enlarged which leads to a lower resonance frequency. This phenomenon has to be considered when the dimensions of the cylindrical resonator are designed. A detailed discussion about how the resonance frequency is affected by the inserted quartz tube can be found in Reference 23. If a long and extended quartz tube is used, this can also act as the reaction chamber for the volume processes. However, for surface treatments the plasma can also be shaped differently by different kind of orifices. The microwave is supplied via a rectangular waveguide from the magnetron. To avoid noise nuisance the use of a low ripple magnetron is recommended. The magnetron which is used in the movie is a low ripple one.

For the ignition of the plasma the high quality coaxial resonator is used while a stable and continuous operation is provided by the cylindrical resonator. To achieve the ignition of the

plasma by the high quality coaxial resonator the resonance frequency of this resonator has to perfectly match the frequency of the microwave provided by the used magnetron. Since all magnetrons do not emit their microwave frequency at exactly the nominal frequency and since the frequency is dependent on the output power, the magnetron has to be measured with a spectrum analyzer. The resonance frequency of the coaxial resonator can be adjusted by moving the metallic nozzle up and down. This resonance frequency can be measured and thereby also adjusted to the sending frequency of the used magnetron with a network analyzer. To reach the high electrical field at the tip of the nozzle, required for the ignition of the plasma, a three stub tuner is needed in addition. This three stub tuner is a commonly used microwave component. The three stub tuner is mounted between the microwave plasma torch and the magnetron. After the resonance frequency of the coaxial resonator is adjusted, the forward power is maximized and the reflected power minimized by iteratively adjusting the stubs of the three stub tuner.

After having adjusted the resonance frequency of the coaxial resonator as well as having maximized the forward powers by means of the three stub tuner, the plasma of the microwave plasma torch can be ignited when the microwave plasma torch is connected to a magnetron. For the ignition of the plasma a minimum microwave power of about 0.3 to 1 kW is sufficient. The plasma ignites in the coaxial resonator. After the ignition of the plasma the resonance frequency of the coaxial resonator is shifted due to the dielectric permittivity of the plasma and the microwave can no longer penetrate into the coaxial resonator. Thus, the plasma switches from the coaxial mode into its much more extended cylindrical mode burning freely-standing above the metallic nozzle in the center of the cylindrical resonator. Since the quality of the cylindrical mode is very low and therefore exhibits a broad resonance curve, the microwave can still penetrate into the cylindrical resonator despite of the shift of the resonance frequency due to the dielectric permittivity of the plasma. Thus, a continuous and stable operation of the plasma in the cylindrical mode is provided by the microwave plasma torch. However, to reach a complete absorption of the supplied microwave power, the stubs of the three stub tuner have to be readjusted. Otherwise the supplied microwave power is not completely absorbed by the plasma but some percentage of the provided microwave is reflected and absorbed by the water load.

To examine the ignition of the plasma in the coaxial mode and then its transition into the extended cylindrical mode, the plasma ignition is observed by a high speed camera.

The presented movie will show how the frequency dependence of the magnetron is measured, the resonance frequency of the coaxial resonator is adjusted, how the forward power is maximized and how the plasma is ignited by the supplied microwave power. The high speed camera recording is shown as well as.

PROTOCOL:

1. Measurement of the Magnetron

Note: The schematic of the experimental setup for measuring the magnetron is depicted in Figure 1a.

1.1) Connect the magnetron to an insulator consisting of a circulator and a water load with 10 screws.

1.2) Connect the insulator to a directional coupler with 10 screws.

1.3) Connect the directional coupler to a second water load with 10 screws.

1.4) Supply all water loads with water.

1.5) Calibrate the spectrum analyzer with its calibration function according to manufacturer's protocol.

1.6) Connect a 20 dB attenuator to the spectrum analyzer by plugging the 20 dB attenuator to the spectrum analyzer.

Note: The 20 dB attenuator is used to protect the spectrum analyzer from too high powers above 1 W.

1.7) Connect the 20 dB attenuator equipped spectrum analyzer to the end of the coaxial cable equipped with a BNC connector by plugging the coaxial cable into the 20 dB attenuator.

1.8) Connect the end of the coaxial cable equipped with an N connector to the directional coupler by plugging the coaxial cable to the directional cable.

1.9) Switch on the magnetron via the power supply and the spectrum of the emitted microwave is displayed on the spectrum analyzer.

1.10) If necessary, adjust the displayed abscissa, ordinate and their resolution according to the manual of the spectrum analyzer.

1.11) To measure the frequency of the output microwave in dependence of the microwave power, increase the microwave power from 10% to the maximum of the output power in 5% to 10% steps and for every step determine the frequency of the maximal amplitude of the spectrum displayed by the spectrum analyzer.

Note: Usually, the frequency spectrum of a magnetron below 10% of its maximum output power is very broad, exhibits many different peaks and therefore is not usable.

2. Adjustment of the Resonance Frequency

Note: The schematic of the experimental setup for measuring and adjusting the resonance

frequency is depicted in Figure 2a.

2.1) Calibrate the network analyzer with the calibration kit for S11 operation (according to manufacturer's protocol).

2.2) Connect the coaxial cable via the N-connector to the coaxial part of a coaxial-to-rectangular-wave guide transition by plugging the coaxial cable to the coaxial-to-wave-guide-transition.

2.3) Connect the rectangular part of the coaxial-to-rectangular-wave guide transition to a three stub tuner with 10 screws.

2.4) Connect the three stub tuner to the microwave plasma torch assembly with 10 screws.

2.5) In the network analyzer menu switch to S11 operation.

2.6) In the network analyzer menu switch to VSWR mode or to log mode.

2.7) Iteratively adjust the resonance frequency of the microwave plasma torch assembly to the measured frequency of the magnetron at an output power of 25 - 60% of the maximum output power by moving the nozzle up and down. The resonance frequency of the microwave plasma torch assembly is given by the dip of the S11 parameter measurement as depicted in Figure 2b. Adjust this dip by moving the nozzle up and down to the recommended frequency.

2.8) When the resonance frequency is adjusted, lock the position of the nozzle with the locking nut.

2.9) Increase the forward microwave power iteratively by adjusting the three stubs of the three stub tuner by moving the stubs up and down. The microwave power absorbed by the microwave plasma torch assembly is given by the depth of the dip of the S11 parameter. Thus, maximize this dip by adjusting the stubs of the three stub tuner. Commonly, it is sufficient that two of the three stubs are used.

3. Ignition of the Plasma

3.1) Wear UV protection glasses since the plasma emits UV radiation. Operate the plasma torch under local gas ventilation since the plasma produces nitride oxides.

3.2) Connect the microwave plasma torch assembly with the adjusted coaxial resonator (nozzle is locked) and the adjusted three stub tuner to the magnetron equipped with an insulator consisting of a circulator connected to a water load.

3.3) Connect the gas supply to the microwave plasma torch.

3.4) Turn on the gas supply to 5 to 20 slm.

3.5) Since microwave radiation in higher doses is harmful especially for the eyes, check that there are no microwave leakages.

3.5)1. To do so, turn on the microwave at a very low power of 10 to 12% and check all microwave connections with a microwave meter for leakages.

3.5)2. If there are any leakages remove them completely before increasing the microwave power or operating the microwave plasma torch.

3.6) If there are no leakages turn on the microwave starting with low powers of 10% and increase the microwave power slowly within 10 to 60 s until the plasma ignites in the quartz tube of the microwave plasma torch.

3.7) Carefully observe if and where the plasma ignites but be careful with possibly radiated microwaves. Preferably use a mirror for the observation of the plasma ignition.

3.8) If no plasma ignites, switch off the microwave power and carefully check if the microwave power is properly coupled into the coaxial resonator and not misguided to other components heating them up or even harming them. Check if some components are getting heated up.

3.8)1. If any component gets heated up – i.e., the microwave power is misguided – move all stubs of the three stub tuner out of the waveguide and adjust them to maximize the microwave coupling into the plasma torch assembly as described in step 2.9. Then start again with step 3.1.

3.8)2. Adjust the resonance frequency of the coaxial resonator of the plasma torch to the sending frequency of the magnetron at a high enough microwave power output of 25 to 60% of the maximum output power with the network analyzer as described in step 2. To improve the ignition, adjust the resonance frequency of the coaxial resonator as described in step 2 to a higher output power. Then start again with step 3.1.

3.9) If the plasma ignites somewhere in the plasma torch and does not automatically switch to the coaxial or cylindrical mode, vary the supplied microwave power and gas flow until it burns in the cylindrical mode.

3.10) When the plasma burns in the cylindrical mode, iteratively adjust the stubs of the three stub tuner by moving them up and down so that all of the supplied microwave power is absorbed by the plasma and the reflected microwave power becomes zero.

Note: If a microwave diode is connected to the water load and to the corresponding input of the control unit, the reflected microwave power is displayed at the control unit of the microwave power supply. How to do this is described in the manual of the microwave power

supply.

3.11) When higher microwave powers of 1.5 kW or more and low gas flows of less than 15 slm are used, check carefully that the plasma does not touch the walls of the quartz tube. The quartz tube must not glow anywhere.

3.12) If the quartz tube glows red, decrease the microwave power or increase the gas flow until it vanishes completely.

3.13) Since microwaves can be radiated by the plasma due to the conductivity of the plasma, check with a microwave meter that the radiated microwave power is below the threshold.

3.14) If the radiated microwave power is above the threshold, shield the plasma with a metallic mesh where the mesh size is much smaller than half of the used microwave wave length.

4. High-Speed Camera Movie of the Plasma Ignition

Note: Since the ignition of the plasma and its transition to the cylindrical mode is in the range of some hundred milliseconds, this process can best be investigated by means of a high speed camera. However, it is not necessary to observe the ignition process by means of a high speed camera each time the plasma is ignited.

4.1) Place the lens of the high speed camera in front of the microwave plasma torch looking through the diagnostic slit at the front of the plasma torch.

4.2) Adjust until the camera is pointing into the coaxial resonator at the tip of the metallic nozzle.

4.3) Focus the camera on the tip of the metallic nozzle.

4.4) Start the recording with 1000 fps (frames per second) of the high speed camera.

4.5) Ignite the plasma as described in section 3.

5. Stable and Continuous Plasma Operation

Note: When the plasma has been ignited in the cylindrical mode and the three stub tuner has been adjusted to maximize the absorption of the microwave power by the plasma a stable and continuous operation of the plasma torch is possible.

5.1) Adjust the dimension – the radial and axial extension – of the plasma to the desired dimension by varying the supplied microwave power between 10% and the maximum output power and the gas flow between 10 and 70 slm. Keep the radial dimension limited to the

diameter of the quartz tube. The plasma must not touch the wall of the quartz tube which means that the quartz tube must not glow.

5.2) To shape the plasma to different shapes, use a short quartz tube which only confines the plasma inside of the cylindrical resonator and place one orifice on the top of the plasma torch assembly.

5.3) If necessary, fasten the orifices with some screws.

REPRESENTATIVE RESULTS:

To provide a plasma ignition without any additional igniters as well as a stable and continuous plasma operation a high quality coaxial resonator with an adjustable resonance frequency was combined with a low quality cylindrical resonator to a microwave plasma torch. The schematic of this plasma torch is presented in Figure 3. The plasma is confined into a microwave-transparent tube, here a quartz tube. This tube can act as a reaction chamber for volume plasma processes or a plasma brush for surface treatments can be formed by an orifice. The microwave power is guided via a rectangular waveguide from the magnetron to the microwave plasma torch. Different kinds of gases can be supplied via either the tangential gas supply or axially through the metallic nozzle of the coaxial resonator. The microwave plasma torch is equipped with a frontal slit, so that the plasma inside the torch and the ignition can be investigated in detail.

To guarantee an ignition of the plasma solely by the supplied microwave power a high electrical field of about 3 to 6 MV/m is needed. To get a better understanding of the electrical field distribution, simulations of the electric field distribution as well as Eigenmode analysis with the commercially available simulation software COMSOL Multiphysics were conducted. Modeling and simulations of electrical field distributions of atmospheric pressure microwave plasma torches provided already detailed insights and led to further developments and improvements regarding for example their ignition or operation behavior^{19 – 22}.

The electrical field distribution of the coaxial mode as well as of the common cylindrical E_{010} mode is depicted in Figure 4a and 4b, respectively. The electrical field is displayed in arbitrary units, since the electrical field in the coaxial resonator is many times higher compared to the electrical field in the cylindrical resonator. It can be seen that a high electrical field at the nozzle tip is reached with the coaxial resonator and the highest electrical field of the cylindrical resonator is in the center of the cylindrical resonator. The resonance frequency of the coaxial resonator can be varied by the position of the metallic nozzle. The simulation results for the resonance frequencies for different nozzle positions for a microwave plasma torch with a cylindrical resonator with a radius of 0.05 m and a height of 0.048 m are shown in the diagram in Figure 4c. It can be seen that the resonance frequency of the cylindrical mode is not affected by the position of the metallic nozzle. However, the resonance frequency of the coaxial mode is dependent on the nozzle position and decreases when the metallic nozzle is moved upwards into the cylindrical resonator.

To reach the required high electrical field in the coaxial resonator this resonance-frequency-adjustable coaxial resonator exhibits a high quality and a sharp and narrow resonance curve. However, a sharp and narrow resonance curve requires that the resonance frequency of the coaxial resonator matches perfectly the frequency of the supplied microwave. Since usually magnetrons do not emit the microwave at their nominal frequency and since the frequency of the microwave is dependent on the output power of the microwave, the frequency dependence of the magnetron has to be measured by means of a directional coupler and a spectrum analyzer. The experimental set-up to measure the frequency dependence of the magnetron with a spectrum analyzer is schematically given in Figure 1a. The measured frequency dependence of the utilized magnetron is shown in the diagram in Figure 1b. The center frequency was set to 2.45 GHz and the video bandwidth was 200 MHz. It can be seen that at a power of 200 W (10% of the maximum output power of the magnetron) the frequency of the microwave is at 2.44638 GHz and increases when the microwave power is increased. At the maximum output power of 2 kW the microwave frequency reaches a value of 2.45213 GHz.

The resonance frequency of the microwave plasma torch can be measured with a network analyzer and since the nozzle is movable the resonance frequency of the coaxial resonator can be adjusted. To do so, the microwave plasma torch assembly has to be connected to a network analyzer via a rectangular-to-coaxial waveguide transition to the network analyzer as shown in the schematic in Figure 2a. By measuring the S11 parameter of the microwave plasma torch assembly the resonance frequency can be determined. The S11 parameter represents the ratio of the input power to the reflected power in dependence of the frequency. When a resonance is reached, an electrical field establishes in the resonator structure leading to a reduced reflected microwave power. However, the field strength inside the cavity is directly related to the fixed wave amplitude of the microwave provided by the network analyzer. A dip appears in the S11 spectrum which corresponds to the resonance frequency. A typical measurement of the S11 parameter is depicted in Figure 2b. Here a resonance is observed at a frequency of 2.846 GHz. By moving the metallic nozzle up and down, the resonance frequency of the coaxial resonator can be varied as the simulations depicted in Figure 4c showed. This dependence of the resonance frequency of the coaxial resonator on the metallic nozzle position can be measured by means of the S11 parameter. A measurement of the resonance frequency in dependence of the nozzle position and the appertaining simulation results are presented in the diagram in Figure 2c. This diagram shows that there is a good agreement between the simulation results and the measured values of the resonance frequency. The very small shift of the two curves can be explained by very small deviations of the geometry or dimension of the manufactured nozzle compared to the ones used for the simulations. To adjust the resonance frequency of the coaxial resonator to the frequency of the supplied microwave, the metallic nozzle has to be iteratively moved up and down until the dip in the S11 parameter is located at the measured microwave frequency. Then the metallic nozzle has to be locked and the forward power can be maximized by iteratively adjusting the stubs of the three stub tuner so that the S11 parameter dip reaches its maximum depth. The high quality of the resonator and the maximized forward power lead to fewer microwave reflections and a high electrical field is established in the resonator which is why a deep dip in the S11 parameter results.

After the microwave plasma torch assembly is mounted to the magnetron and the gas supply is connected, the plasma torch can be ignited and operated. The ignition of the plasma can be investigated best by observing the ignition with a high speed camera. The ignition of the plasma was recorded at 1000 fps. The presented plasma ignition was conducted at a microwave power of 1 kW and a supplied gas flow of 15 slm air. Images of each phase of the ignition are summarized in Figure 5. The image in Figure 5a shows the view from above, looking down on the nozzle at an angle through the diagnostic slit at the front of the inoperational plasma torch. The bottom of the cylindrical resonator is in the front. In the mid plane you can see the beginning of the coaxial resonator. The tip of the nozzle can also be seen. The bottom of the cylindrical resonator is located in the background again. Since the focus is on the nozzle tip, the bottom of the cylindrical resonator is somewhat blurry. The other images show the phases of the plasma ignition. When the microwave power is turned on at $t = 0$ ms, the plasma ignites somewhere in the coaxial resonator as can be seen in Figure 5b. Then, during 64 ms, the plasma winds up the metallic nozzle to its tip and then burns straight at the nozzle tip in the coaxial mode as the Figures 5c to 5e show. The intensity of the plasma grows for the following 692 ms as it is shown in Figure 5f. Then, due to the shift of the resonant frequency caused by the burning plasma in the coaxial resonator one millisecond later, the plasma starts to break away from the nozzle tip as shown in Figure 5g and 5h. The complete break away of the plasma from the nozzle tip is reached after 58 ms as depicted in Figure 5i. The plasma is now burning freely above the metallic nozzle in the cylindrical mode. During the last second, the three stub tuner is readjusted to maximize the forward microwave power. This leads to an increase of the plasma as the image in Figure 5j shows. However, the plasma is still burning freely above the nozzle tip with no contact to it. Due to the low quality of the cylindrical resonator the plasma can be operated continuously and stably in this cylindrical resonator mode.

The dimension of the plasma depends on the supplied microwave power and the gas flow. Photos of the plasma for microwave powers of 1 and 2 kW and gas flows of 10, 30 and 70 slm are presented in Figure 6. The resonator with its diagnostic slit at its front is located in the lower part of the photos. The plasma is confined into a quartz tube within and above the cylindrical resonator. UV light couples into the quartz tube which is why the quartz tube exhibits a bluish glowing. It can be seen that the dimensions – radial and also the axial extension – of the plasma increase with an increase of the supplied microwave power while an increase of the gas flow leads to a smaller plasma flame. However, measurements of the gas and electron temperature show the maximum temperatures of $T_g = 3600$ K and electron temperature $T_e = 5800$ K are independent of the outer parameters, supplied microwave power and gas flows, as well as of the plasma volume¹⁹. The temperatures were obtained by means of optical emission spectroscopy. The $A^2\Sigma^+ - X^2\Pi_\gamma$ -transition of the free OH radical was used for the determination of the gas temperature while a Boltzmann-plot of atomic oxygen lines was conducted for the estimate of the electron temperature. A detailed description on how the temperatures have been measured and the complete temperature distributions can be found in References 23 and 24.

To treat surfaces in the afterglow of the plasma, the plasma can be shaped with different kinds of orifices. Figure 7 depicts photos of differently shaped plasmas. The layout is similar to the

photos of the plasma confined to a long quartz tube: the cylindrical resonator is at the bottom of the image; its diagnostic slit illuminated by the plasma. Differently shaped plasmas can be seen burning above the top-opening. On the photo in Figure 7a the confining quartz tube does not extend outside of the resonator. The plasma can burn freely above the resonator. An extended plasma brush can be formed with a slit orifice as depicted in Figure 7b. A plasma needle can be achieved by using an orifice with a hole in its center. This is shown in Figure 7c. Very small and smooth afterglow plasmas are formed by orifices which have a narrow slit or some small holes arranged in a circle as the photos in Figure 7d and 7e show.

Figure 1: Measurement of the magnetron. The schematic in a) shows how the frequency dependence on a magnetron of the microwave output power can be measured by means of a spectrum analyzer. The frequency dependence on the used magnetron of the output power is depicted in b)

Figure 2: Coaxial and cylindrical mode. The distribution of the electrical field strength is depicted in a) and b). a) shows the distribution for the coaxial mode while b) shows the one for the cylindrical mode. The diagram in c) shows the dependence of the resonance frequency of both the coaxial and the cylindrical mode on the position of the metallic nozzle in the plasma torch. The resonator has a diameter of 0.05 m and a height of 0.0482 m.

Figure 3: Plasma torch setup. Schematic of the setup of the atmospheric microwave plasma torch.

Figure 4: Measurement of the resonance frequency. The setup for the measurement and adjustment of the resonance frequency of the microwave plasma torch by means of a network analyzer is given in a). b) shows a typical measurement of the S11 parameter. The dip in the S11 parameter reflects the resonance frequency of the microwave plasma torch. The measured dependence of the resonance frequency on the metallic nozzle position and the results of the numerical simulations are summarized in c).

Figure 5: Ignition of the plasma. Images of each phase of the ignition of the plasma recorded by a high speed camera at 1000 fps and at a microwave power of 1 kW and a gas flow of 15 slm air. a) view from above, looking down on the nozzle at an angle through the diagnostic slit at the front of the inoperational plasma torch. b) ignition of the plasma in the coaxial resonator. c) to e) winding up of the plasma to the tip of the metallic nozzle until it burns in the coaxial mode. f) the plasma increases. g) to i) the plasma breaks away from the metallic nozzle and burns freely above the nozzle tip in the cylindrical mode. j) the plasma increases due to the readjustment of the three stub tuner to maximize the forward power.

Figure 6: Photos. Photos of an air plasma for different supplied gas flows of 10, 30 and 70 slm and microwave powers of 1 and 2 kW. The extent of the plasma depends on the supplied microwave power and gas flow and it increases with an increase of the microwave power and a decrease of the gas flow.

Figure 7: Different orifices. By using differently shaped orifices the plasma can be formed. a) the confining quartz tube does not extend outside of the resonator and the plasma can burn freely above the resonator. b) the plasma is shaped into a brush with a slit orifice. c) a plasma needle is formed by a hole orifice. d) a very smooth plasma brush can be achieved by using an orifice with a narrow slit and e) a smooth plasma area is formed by an orifice with some small holes arranged in a circle.

DISCUSSION:

The presented movie explains how an ignition of an atmospheric pressure microwave plasma without any additional igniters can be realized, the basic principles of this microwave plasma torch, its adjustment, the ignition process of the plasma and its stable and continuous operation. As described in the introduction, there already are different kinds of microwave plasma torches but none of those provide an ignition of the plasma without any additional igniters as well as stable and continuous plasma operation.

To obtain an ignition of the plasma without any additional igniters at atmospheric pressure a high electrical field is necessary and therefore a resonator with a high quality while for a continuous and stable plasma operation a low quality is needed. This can be realized by combining a high quality coaxial resonator which guarantees the ignition of the plasma and a low quality cylindrical resonator which provides a continuous and stable plasma operation.

The frequency of the supplied microwave has to perfectly match the resonance frequency of the high quality coaxial resonator so that the provided power is coupled into the resonance chamber. Therefore the frequency dependence of the magnetron has to be well known and the resonance frequency of the coaxial resonator has to be adjustable. The sending frequency of the magnetron can be measured with a spectrum analyzer while the resonant frequency of the coaxial resonator can be measured by means of a network analyzer and adjusted by the movable nozzle.

To guarantee the ignition of the plasma solely by the supplied microwave, it is crucial that the resonance frequency of the coaxial resonator perfectly matches the sending frequency of the magnetron. Furthermore, the microwave has to be coupled completely into the coaxial resonator of the plasma torch assembly which is achieved by maximizing the forward power with the three stub tuner. If these critical steps are not conducted carefully it is possible that the plasma will not ignite or that the microwave is coupled into the experimental setup somewhere what could lead to some damage of these parts. Thus if no ignition of the plasma is observed, these steps have to be checked carefully again. Furthermore, it is possible that the plasma ignites but does not switch to the coaxial or cylindrical mode by itself. In this case the plasma can commonly be switched first to the coaxial mode and then to the cylindrical mode by varying the gas flow and the supplied microwave power.

To obtain a more automatic ignition and operation of the plasma an automatic three stub tuner which automatically adjusts its stubs to maximized forward power can be used instead of the manual one. Thus the adjustment of the stubs for ignition of the plasma and afterwards the

adjustment for the operation of the plasma are automatically conducted by this three stub tuner. To achieve plasma ignition without any additional igniters and stable and continuous plasma operation the presented smart combination of the two resonator structures and the presented technique of the measurement of the magnetron by a spectrum analyzer and the measurement and adjustment of the resonant frequency by means of a network analyzer are crucial.

The ignition of the plasma was investigated in detail with a high speed camera. It revealed that the plasma ignites in the coaxial resonator, winds up to the tip of the nozzle burning in the coaxial mode, increases in intensity and volume, breaks away from the metallic nozzle, increases further and then burns freely above the metallic nozzle in the cylindrical mode. After the ignition of the plasma and its transition to the cylindrical mode the plasma can be operated stably and continuously. The dimension of the plasma depends on the supplied microwave power and gas flow and increases when the supplied microwave power is increased or the gas flow is decreased. Furthermore, the plasma can be shaped to needles, brushes or smooth afterglow plasmas by using orifices.

The gas flow and the microwave power of the presented microwave plasma torch are limited to about 100 slm and some kW which also limits the volume of the plasma. Since the quartz tube must not be damaged the radial diameter of the plasma is limited to the inner diameter of the quartz tube. If a larger plasma volume is required or large gas flows have to be treated, the plasma source can be up-scaled by using a lower microwave frequency, for example 915 MHz instead of 2.45 GHz. With 915 MHz more microwave power is available, leading to larger plasma volumes which allow larger gas flows to be handled. However, when higher powers are used, the risk of damage, especially of the metallic nozzle, during the ignition of the plasma or during operation increases and therefore another ignition mechanism has to be considered. Furthermore, the plasma parameters, like electron and gas temperature, are independent of the outer parameters like gas flow and supplied microwave power. Thus, if an atmospheric pressure plasma with different plasma parameters is needed, a different source has to be used or one which meets the required needs has to be newly developed.

Since the presented atmospheric pressure microwave plasma torch provides ignition of the plasma without any additional igniters as well as stable and continuous plasma operation, the plasma source is suitable for many industrial applications. The advantage of the ignition of the plasma without any additional igniters for industrial processes, especially when an automatic three stub tuner is used, is that only the microwave has to be switched on and the process starts to run reliably and automatically. Furthermore, if a discontinuous operation is needed where the process is running for some time followed by intermittency, the plasma process can be restarted quickly, reliably and automatically and there is no attrition of an additional ignition system. Volume processes like chemical synthesis as well as surface treatments with the afterglow plasma can be named as applications of the microwave plasma torch. Studies on the successful degradation of harmful waste gases, especially for greenhouse gases like perfluorinated compounds which are used in the growing semiconductor industry, on the dissociation of CO_2 to CO and O as well as on the pyrolysis of methane to hydrogen and carbon

have already been conducted. Furthermore, the afterglow plasmas were used for the treatment of surfaces to increase the adhesion of glue and paint and for decontamination and sterilization purposes. For example, the plasma source can be used for the decontamination of the surface of cork stoppers to degrade trichloroanisole, which causes the so called cork taint. Another application is the reduction of germs on surfaces, like on packaging materials or on food.

The presented technique how the sending frequency of a high frequency power supply is measured by means of a spectrum analyzer and how the resonant frequency of a resonant structure is measured and adjusted by means of a network analyzer can also be applied to other high frequency plasma sources. As an example a tiny little micro microwave plasma jet which is based on a $\lambda/4$ -resonator can be named^{25 - 27}.

Lastly, the presented movie will lead to further developments and improvements of atmospheric pressure and/or microwave plasma sources.

DISCLOSURES:

The authors have nothing to disclose.

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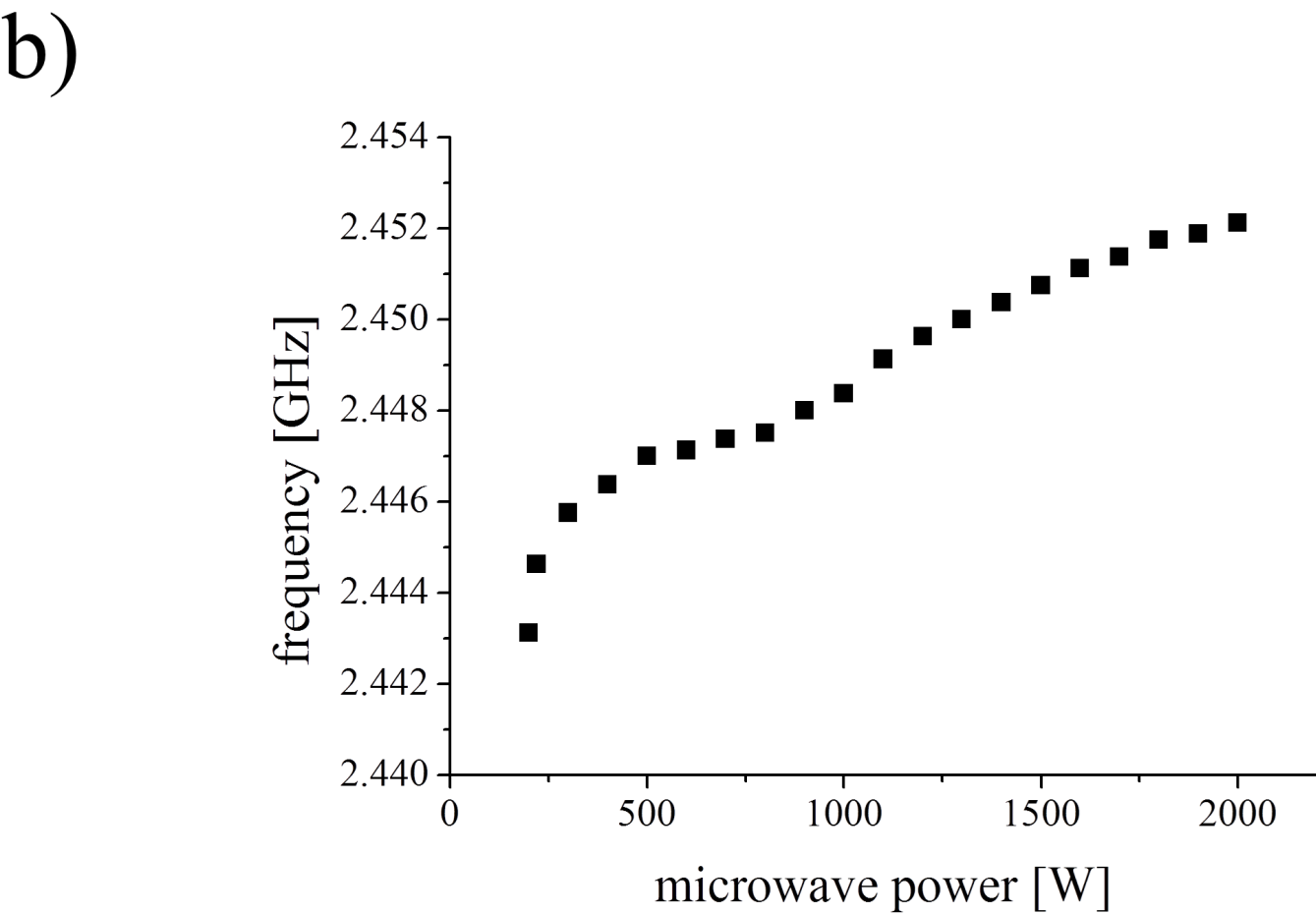
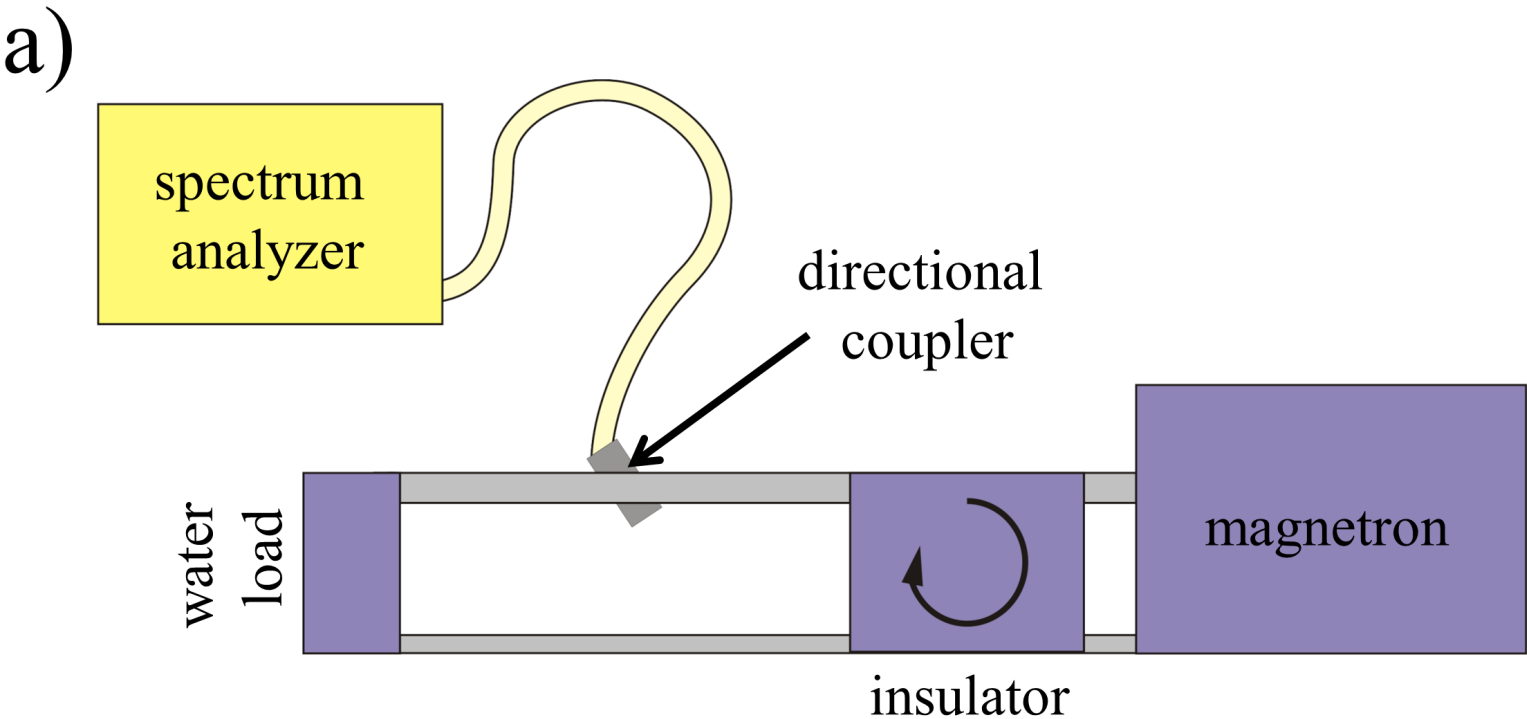
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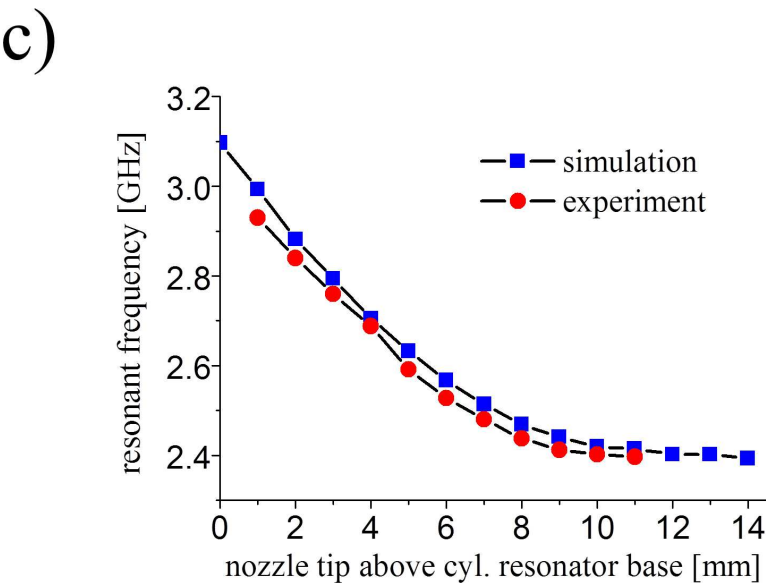
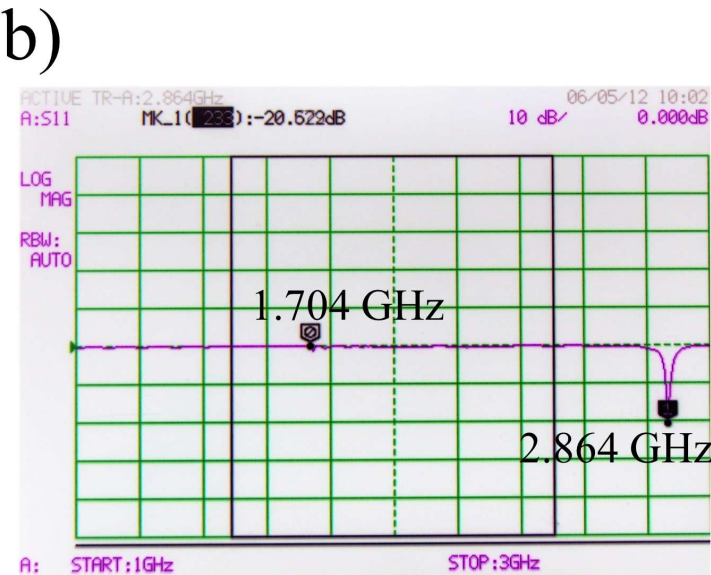
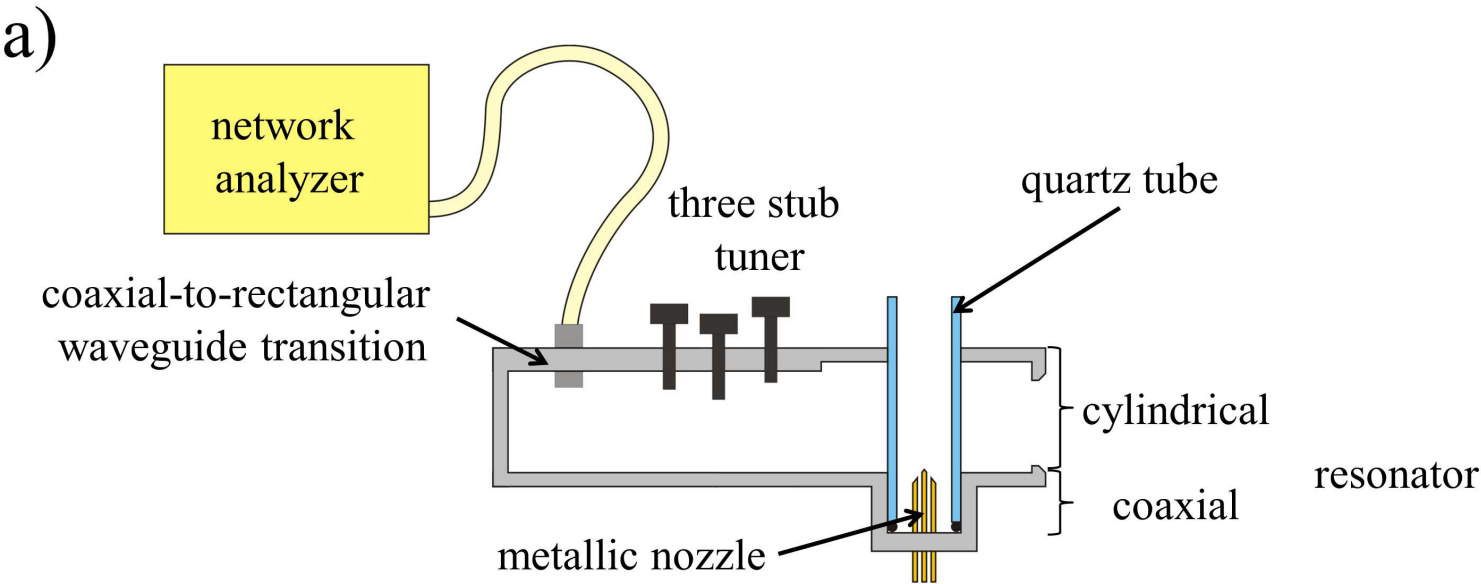
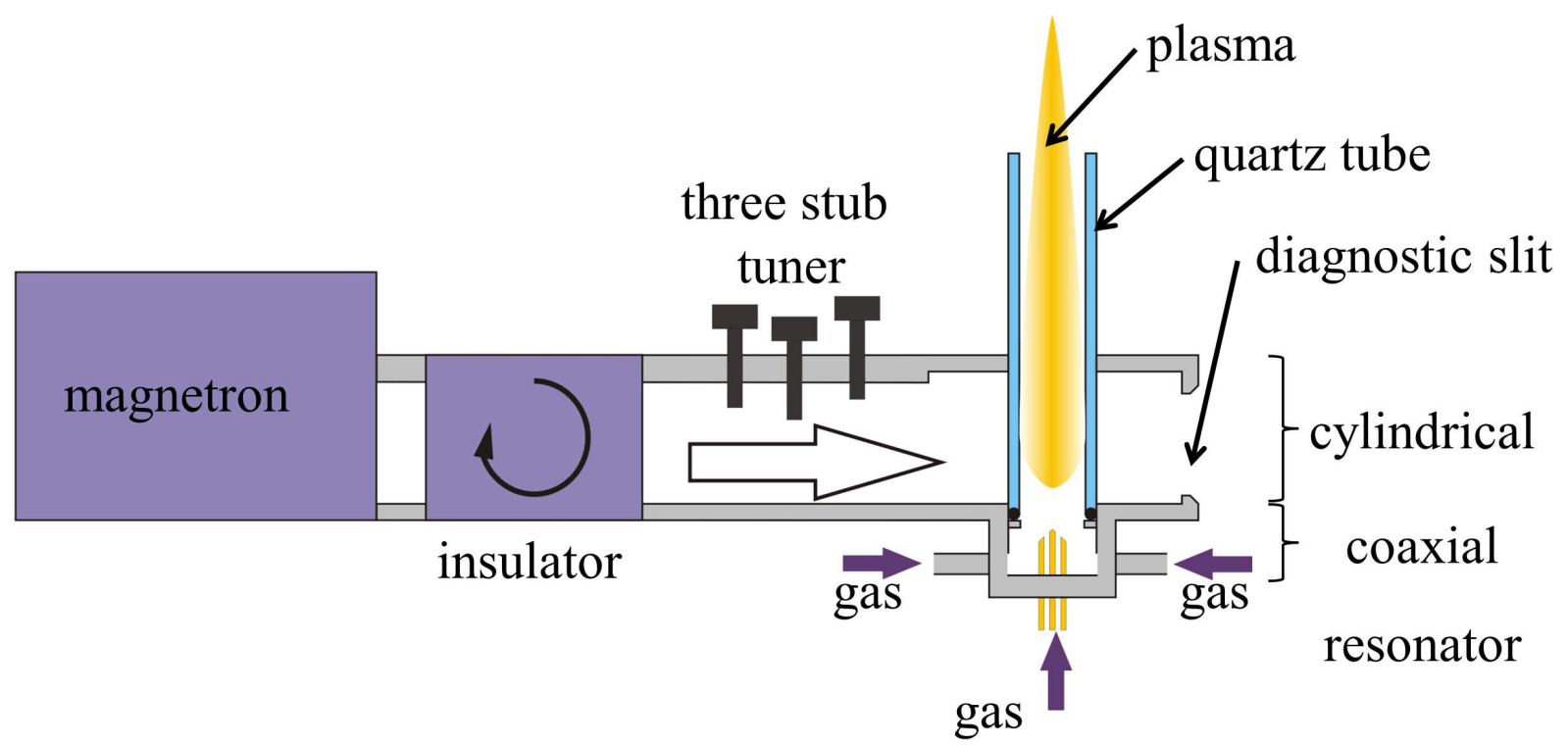
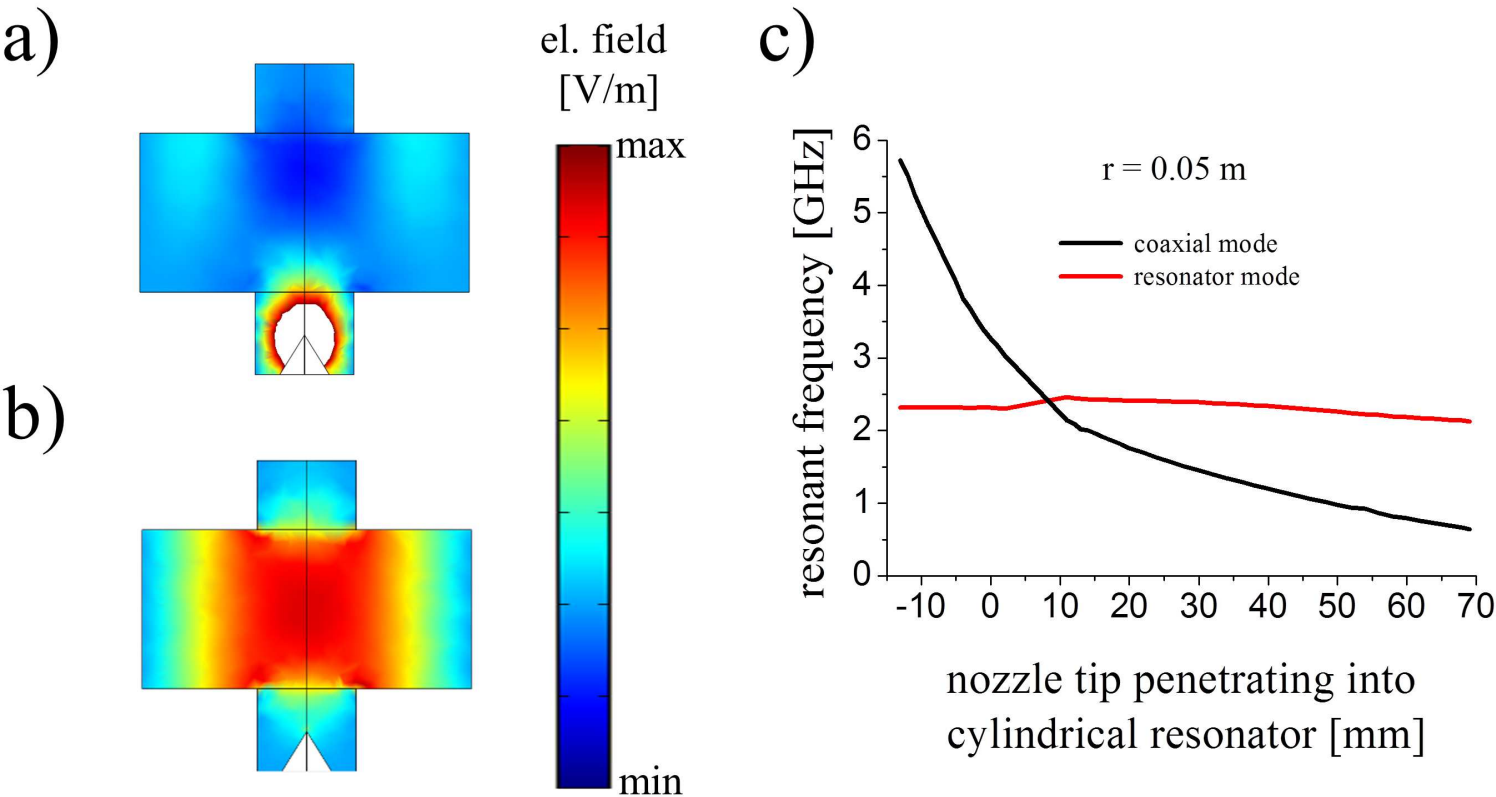
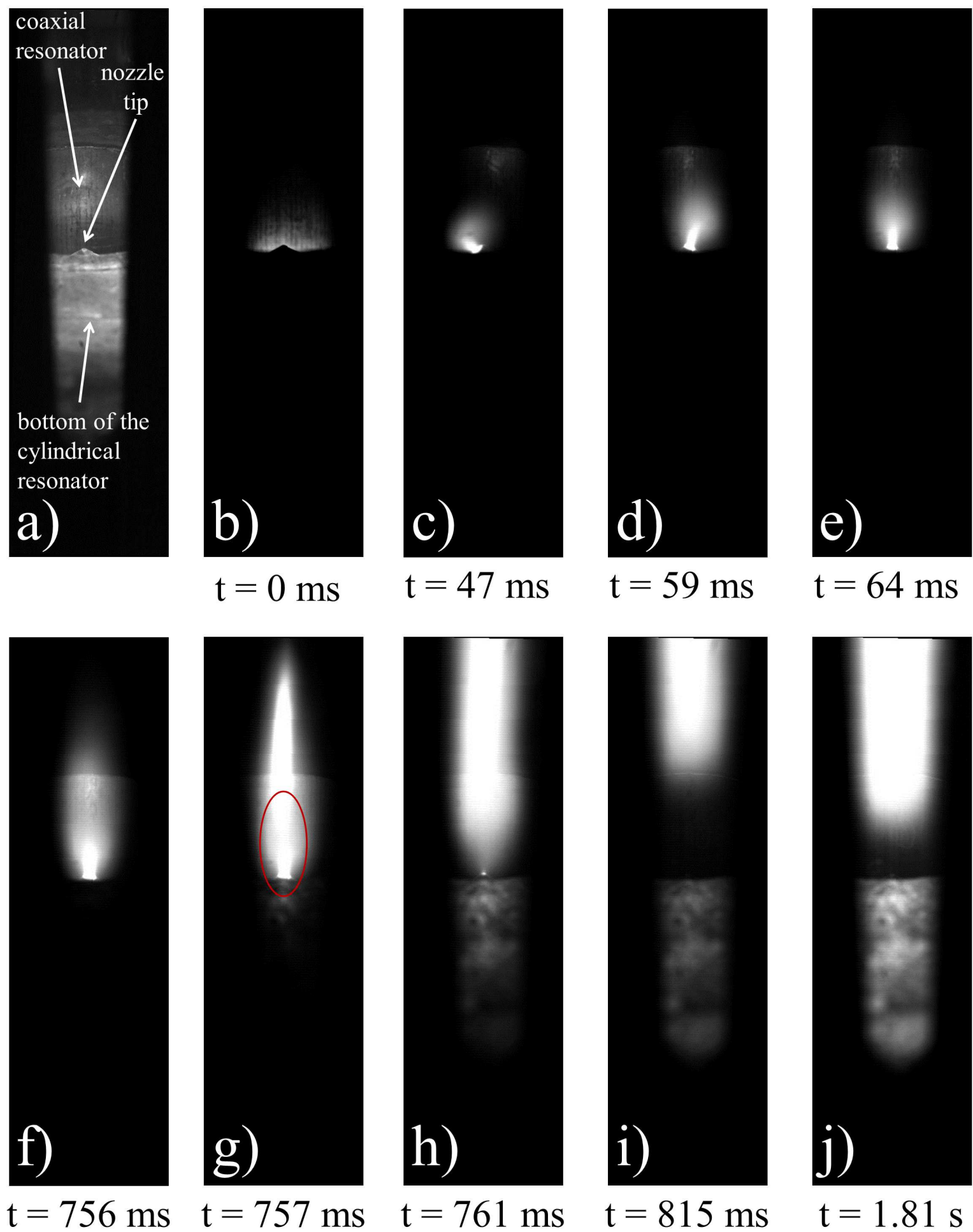
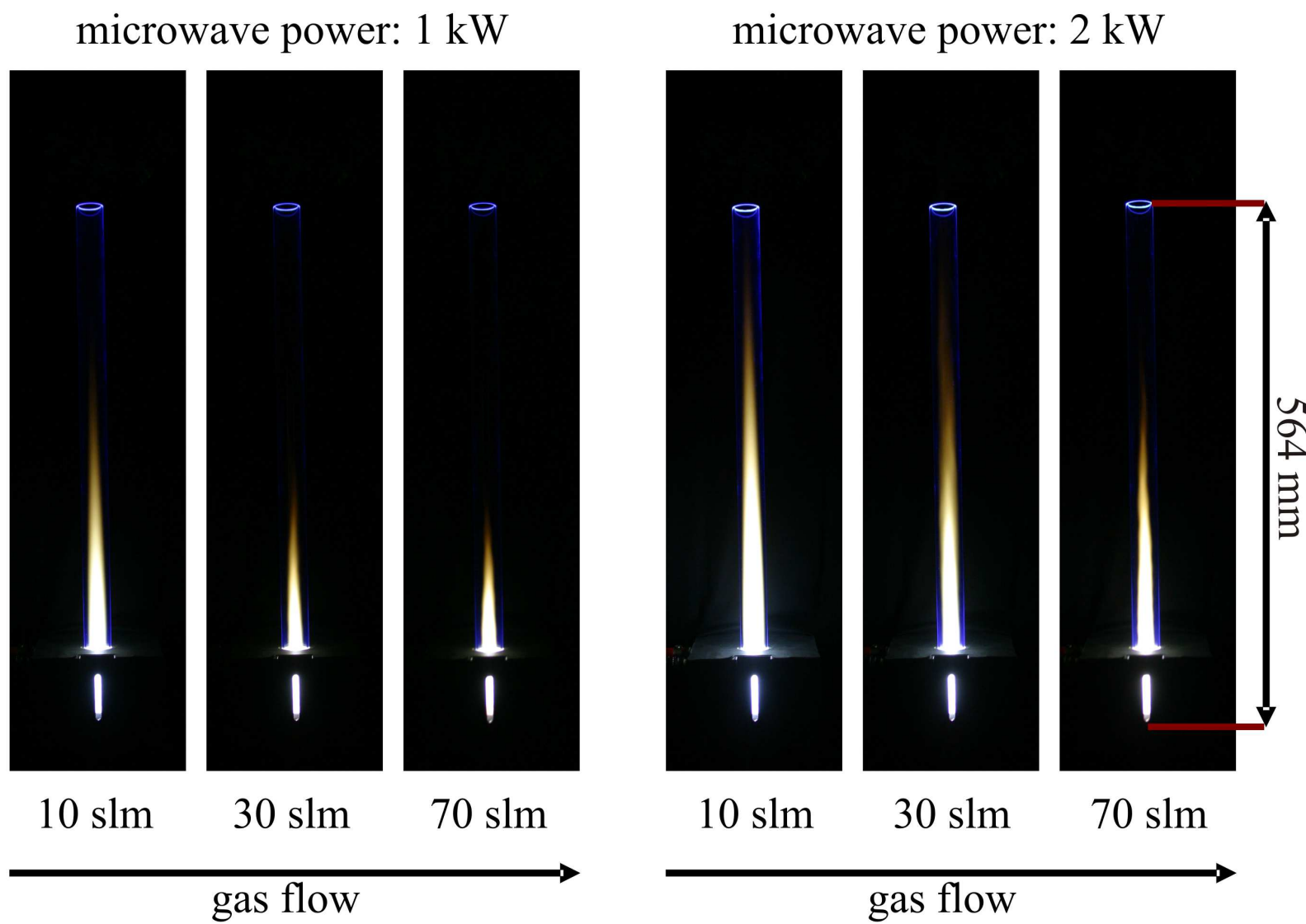


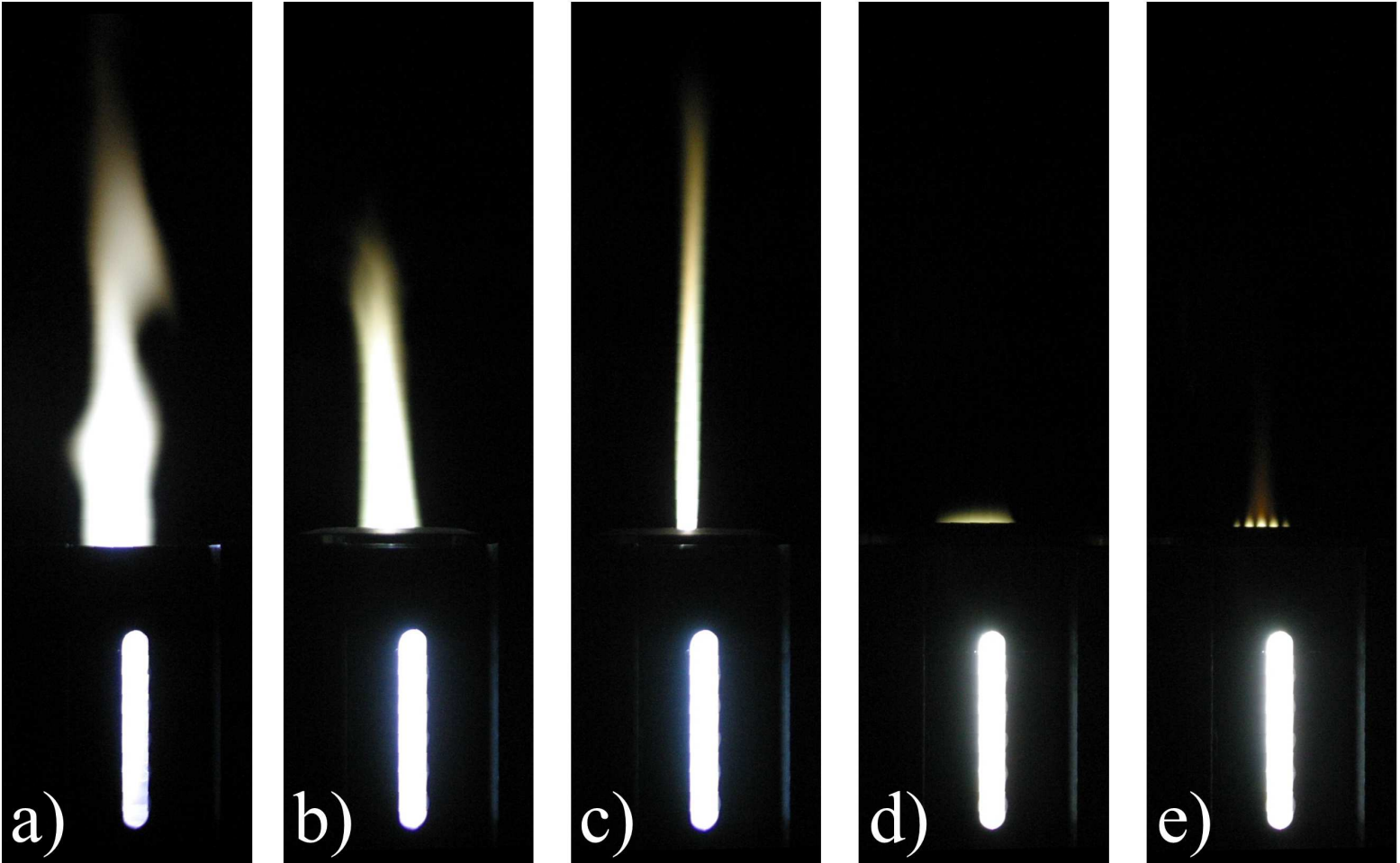
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Name of the Material/Equipment
2 kW magnetron
2 kW power supply
insulator - circulator with water load
water load
three stub tuner
orifices
microwave plasma torch
spectrum analyzer
network analyzer
calibration kit
directional coupler
20 dB attenuator
coaxial to rectangular wave guide transition
adaptor 7-16 to N connector
coaxial cable
high speed camera
lens
local gas ventilation
UV protection glasses
microwave leakage tester
microwave survey meter

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Article Title: How to Ignite an Atmospheric Pressure Microwave Plasma Torch without any Additional Igniters
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Dear Dr. Nguyen,

please find below responds to every recommended comment. The responds to the comments have been written in red below each comment. I have tracked all changes in my word processor, however please note that the given line numbers correspond to the document where all changes have been accepted.

Changes to be made by the Author(s):

1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues. The JoVE editor will not copy-edit your manuscript and any errors in the submitted revision may be present in the published version.

The manuscript has been carefully proofread.

The manuscript will greatly benefit from copyediting by a native English speaker. There are numerous copy-editing errors throughout.

The manuscript has been copy-edited by a native speaker.

2. In Steps 1&2-please add more detail on how things are connected to each other for the narration.

More details on how the mentioned things are connected to each other have been inserted.

3. Please make sure all references are in the correct format (eg. Ref #4 is not).

[LastName, F.I., LastName, F.I., LastName, F.I. Article Title. Source. Volume (Issue), FirstPage – LastPage, doi: DOI (YEAR).]

The references have been reviewed and where needed corrected or completed. However, not for all references all issues like page number, doi, etc. where available. Furthermore, some new references have been added due to reviewer's advices.

Reviewers' comments:

Reviewer #1:

The article provides a very detailed description of a new atmospheric pressure plasma torch and its operation.

Major Concerns:

N/A

Minor Concerns:

N/A

Reviewer #2:

The authors consider and discuss the operation of an atmospheric pressure microwave plasma torch. The research group in Stuttgart is working successfully since many years on microwave plasmas for process technology. Now, they discuss in the submitted paper especially the ignition of such a plasma source.

According to the scope and outline of the journal (JoVE) the authors carefully describe the methods and procedure of their experimental investigations. The recipe-like description of the several steps in the protocol is clear and understandable.

In the following I list some remarks and suggestions for changes / additions:

The authors state that the coaxial resonator exhibits a high quality (line 108). This statement should be explained in more detail, e.g. the effect of the resonator on the plasma operation and the applications might be extended.

In line 108 to 116 more and detailed information is provided now. Furthermore, in line 146 to 162 this issue is discussed, too.

The sequence of the figures starts with Fig.3a (line 159). Fig.s 1 and 2 are mentioned later. The sequence should be corrected.

The sequence of the figures has been corrected.

Concerning the COMSOL Multiphysics simulation (mentioned in line 349) the authors may provide some related references and comparison with MW simulation results of other groups.

In line 379 to 382 references have been inserted. However, a detailed comparison of the presented simulations with results from other groups is not feasible since different geometries have been used.

In lines 436 to 439 the authors list the measured gas temperature and electron temperature, respectively, in the plasma source. How did they obtain these values? Please, describe the used diagnostics and data evaluation in more detail.

In line 476 to 481 more information about the method how the temperatures were measured and also reference where they are described in more detail are given. A more detailed description in this JoVE paper would be beyond the scope of the paper.

Either in the "Introduction" or in the "Discussion" the applications of the MW plasma torch might be discussed more extensively. What are the special purposes of the source the authors studied? In particular, what is the advantage of their findings in regard to the ignition for technological applications?

In line 605 to 622, in the discussion, more information addressing the mentioned issues has been inserted. However, a more detailed discussion about the applications of the plasma source would be beyond the scope of this paper.

Reviewer #3:

Manuscript Summary:

The methods for tuning the double-resonator based atmospheric pressure microwave plasma torch are described in detail so that interested plasma scientists will most probably not face any difficulties to get a similar atmospheric pressure microwave plasma torch in operation without additional igniters. Particularly the video recorded by means of the high speed camera will be of great interest with regard to understanding the single processes from ignition of the plasma in the high-quality coaxial resonator to the plasma finally detaching from the tip of the metallic nozzle. However, it is recommended to the authors to perform some modifications in their paper with regard to English syntax and spelling (see below).

- The title and the abstract are appropriate for this method article.

- The authors provided several examples of application of the atmospheric pressure microwave plasma torch.
- It could not be realized that any major material or equipment would be missing in the table.
- The steps listed in the procedure are described very detailed, so it is expected that the procedure will lead to the described outcome.
- The steps listed in the procedure are clearly explained.
- It does not seem any important step from the procedure missing.
- The authors mention several details that have to be paid particular attention to when reproducing the methods for getting the atmospheric pressure microwave plasma source into operation without any additional igniters.
- The authors revealed all critical steps in detail, so there does not seem any important information missing.
- The anticipated results seem to be reasonable. Consequently, they will be very useful for scientists interested in operation of atmospheric pressure plasma sources without any additional igniters.
- The given references provide a substantial overview on atmospheric pressure plasma sources and their applications and will therefore be very useful for readers.

Major Concerns:

In the paragraph describing the figures 5a) to 5j) (see lines 403 to 427), the cross reference between the text and the figures 5f) to 5j) has to be corrected starting from line 417: [...] The intensity of the plasma grows for the following 692 ms as it is shown in Figure 5[f]). Then, due to the shift of the resonant frequency caused by the burning plasma in the coaxial resonator one millisecond later[,] the plasma starts to break away from the nozzle tip as shown in Figure 5[g]) and 5[h]). The complete break away of the plasma from the nozzle tip is reached after 60 ms as depicted in Figure 5[i]). Now the plasma is burning freely above the metallic nozzle in the cylindrical mode. During the last second[,] the three stub tuner is readjusted to maximize the forward microwave power[...]. This leads to an increase of the plasma as the image in Figure 5[j]) shows. [...]

This has been corrected in line 441 to 464.

Minor Concerns:

I do not have any major concerns with regard to contents. However, the spelling and the syntax have to be significantly improved by the authors.

- Example (lines 301-302): [...] However, it is not necessary to observe the ignition process each time the plasma is ignited by means of a high speed camera. (According to the

misleading syntax, it seems that the plasma is ignited by means of a high speed camera. This can be avoided by changing the syntax: [...] However, it is not necessary to observe the ignition process by means of a high speed camera each time the plasma is ignited.)

This has been corrected in line 329 to 330.

- Example (line 520): it should read "[...] the plasma won't ignite [...]" instead of "[...] the plasma wont ignited [...]".

This has been corrected.

- Example (lines 109-110): [...] The plasma is ignited and confined in a microwave transparent [cylinder,] preferably a quartz tube. [...]" (missing word)

This has been corrected.

- Example (lines 100-101): [...] The in this movie presented microwave plasma torch offers the opportunity of an ignition of the plasma solely with the provided microwave power [...] (German syntax instead of English syntax)

This has been corrected.

- The word "slit" can be found several times in the text with wrong spelling "silt" (in lines 343, 431, 444, 447 and 489).

This has been corrected.

- Some words seem to be used in the wrong context, e.g. "color" should most probably read "paint" (see lines 75 and 87), or "supplement[ary]" in the meaning of "surplus" (see lines 70 and 93).

This has been corrected.

- etc.

Additional Comments to Authors:

The authors are kindly asked to have their paper checked by a native English speaker.

The manuscript has been copy-edited by a native speaker.

Reviewer #4:

Manuscript Summary:

In general the topic is clearly presented. Nevertheless, the writing, the numbers and the referencing should be reviewed.

Major Concerns:

Comment 1 (L 114)

The author recommends a low ripple magnetron. Could the author provide any references, how such low ripple tube can be purchased.

In line 127 to 128 a sentence has been inserted, that the magnetron, which used in the JoVE movie, is a low ripple one. The company where this low ripple magnetron can be purchased is given in the table of materials/equipment.

Comment 2 (L 121)

Could the author provide additional references concerning the dependency of the magnetron frequency from the output power.

Sorry, but I found no further literature dealing with this effect. However, we, at our institute, have measured several magnetrons and we observed this phenomenon for all measured magnetrons.

Comment 3 (L 129)

The author should be stricter with using of quality, in means of electrical field amplification, and forwarded power. This subject is well discussed in [1].

1. Rackow, K., et al., Microwave-based characterization of an atmospheric pressure microwave-driven plasma source for surface treatment. Plasma Sources Science & Technology, 2011. 20(3).

In line 142 this has been corrected according to the reviewer's comments.

Comment 4 (L 294)

To shield the microwave, the mesh size should be much smaller than $\lambda/2$.

In line 322 this has been corrected according to the reviewer's comments.

Comment 5 (L 384)

The author explains the dip in the S11 parameter by storing of the energy in the resonator. If this is the case, than the field strength should increase over the time. However this field strength inside the cavity should be directly related to the fixed wave amplitude of the incident wave, provided by the network analyzer.

In line 419 to 421 this has been corrected according to the reviewer's comments.

Minor Concerns:

Comment 6 (L 110)

The author suggests for confining the plasma a quartz tube. However the effect of the tube to the microwave properties of the cavities should be discussed.

In 1189 to 123 more information on this was added and also a reference where this issue is discussed in detail.

Comment 7 (L 122)

The author starts two following sentences with "the resonant frequency".

This has been changed in line 136 to 137.

Comment 8 (L 139)

The author describes the mode switching of plasma source. It would be interesting how the settings of the three stub tuner, optimized for ignition, influence the power coupling in the cylindrical mode.

More and detailed information on this has been inserted in line 158 – 162.

Comment 9 (L 163)

The directivity of the used directional should be described.

The used directional coupler is a homemade one and no directivity was measured nor needed for this purpose since only the frequency dependence of the measured magnetron is of interest and the value of the microwave power is unimportant.

Comment 10 (L 169)

When measuring with a spectrum analyzer, the used video bandwidth should be mentioned.

More and detailed information on this was inserted in line 404 to 406.

Comment 11 (L 172)

Could the author explain the function of the 20 dB attenuator.

An explanation why this 20 dB attenuator is used was inserted in line 194 to 195.

Comment 12 (L 174)

Concerning the coaxial cable, the author should provide a suggestion for suitable connectors.

Information on this has been inserted in line 197 to 201.

Comment 13 (L 311)

Could the author provide a description of fps.

A description of fps was added in line 340.

Comment 14 (L 343, L 431, L 444)

Is the meaning of used word slit, slit?

This was corrected everywhere in the manuscript.

Comment 15 (L 349)

The author refers to the electric field distribution. However, only the absolute value of it is depicted.

The spatial distribution of the stationary electrical field in the resonator structure is given and not an absolute value.

Comment 16 (L 351)

The author refers to the arbitrary units of the electrical field. Could the author provide a normalized scale. Furthermore the region around the tip is out of scale.

The simulations were conducted with an older version of the COMSOL Multiphysics software and with the new version the old models cannot be opened any more. Therefore, these pictures cannot be changed anymore.

Comment 17 (L 357)

Could the author review the dimension of the resonator.

The dimensions of the resonators have been corrected in line 3890.

Comment 18 (L 393)

Could the author comment on possible error sources for the discrepancy between measurement and simulation.

More and detailed information on this was inserted in line 429 to 431.

Comment 19 (L 403-427)

The references to figure 5 and the timescale should be reviewed.

The references and the timescales have been corrected in line 440 to 463.

Comment 20 (L 569)

Could the author give a reference for the mentioned tiny little micro microwave plasma jet.

Three additional references have been inserted in line 627.