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Radio frequency expanding plasmas at low, intermediate, and atmospheric pressure and their applications*

Gheorghe Dinescu‡ and Eusebiu R. Ionita

National Institute for Lasers, Plasma and Radiation Physics, 409 Atomistilor Street, Magurele MG-36, RO 77125, Bucharest, Romania

Abstract: We report on the operation and characteristics of radio frequency (RF) plasma beam sources based on the expansion of the discharge outside of limited spaces with small interelectrode gaps. The appropriate electrode configuration, combined with high mass flow values and appropriate power levels, leads to small- or large-size plasma jets, working stably at low, intermediate, and atmospheric pressures. The sources are promising tools for a wide range of applications in thin film deposition, surface modification, and cleaning, including the case of temperature-sensitive substrates.

Keywords: radio frequency plasma jets; atmospheric-pressure plasma; plasma sources; surface modification; plasma cleaning.

INTRODUCTION

Expanding plasma sources are of great interest from both practical and fundamental points of view. Their advantages reside in the separation of plasma generation and plasma processing zones. These plasmas are usually obtained under high gas-flow conditions. In low-pressure expanding thermal plasmas [1], an equilibrium plasma is generated by a direct current (DC) discharge in a limited-volume, high-pressure zone, and in high-temperature conditions. By expansion out of the discharge zone, at low pressure and at large distances from the expansion nozzle, the plasma cools out and the gas temperature decreases. A strong nonequilibrium character can be noticed supported by the different relaxation times of the excited levels of plasma species. There is a fundamental difference between a nonequilibrium glow discharge and a low-pressure expanded thermal plasma. While in the glow discharges the excitation is carried out by energetic electrons (ionizing plasma sustained by electronic collisions, $T_e \sim$ 1–4 eV) in low-pressure expanding thermal plasmas the excitation is carried out by heavy particles, mostly ions and metastables (recombining plasma, sustained by energy transfer and recombination processes) and the electron temperatures will be low ($T_e \sim 0.2$–0.5 eV) [1]. This example shows that the gas flow can introduce very peculiar nonequilibrium effects. Such effects could even prevail on the equilibration tendency observed with the pressure increase in gas discharges, and indicates a way to design nonequilibrium plasma sources at intermediate, atmospheric, or even supra-atmospheric pressure.

Some significant examples of nonequilibrium expanding plasma jet sources, in which the electrons play the main exciting role, are the “hollow anode”, “constricted plasma source”, and “the plasma pencil”.

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‡Corresponding author: E-mail: dinescug@infim.ro

The "hollow anode" terminology was used in connection with a low-pressure DC discharge with a concave hemispherical cathode and a small hollow anode placed in the center of the hemisphere [2,3]. The gas is introduced through a hole in the cathode. At low pressure and cathode–anode distances large enough, the electrons are focalized by the cathodic fall on the hole of the anodic nozzle, producing a high ionization degree in this region. The gas passing through this region expands as a highly ionized beam. A cylindrical configuration of the hollow anode discharge with enhancement of ionization by the presence of magnetic field was also developed [4]. It was shown [5] that the system can work also with a flat cathode, but with a limited anode surface around the hole, and that the responsible effect for the bright plasma generated around the nozzle is the increase, in such conditions, of the anodic fall. This type of source was denoted as "constricted plasma source" [6], and a series of patents were filled in relation with the respective discharge type [7–9]. Such sources were applied for low-pressure deposition of nitrides involved in optoelectronics applications [10]. Enlarged in length and powered with high-frequency bipolar pulsed voltages, this system led to a low-pressure linear plasma jet [11].

The "plasma pencil" terminology gained notoriety with another construction type [12,13], designed to work at atmospheric pressure. The source is operated by passing a stream of helium gas through a dielectric tube in which the gas flow is restricted by two perforated dielectric disks, separated by less than 1 cm distance. The electrodes are planar circular rings attached to the disks. A cold long plasma beam is launched through the hole of the outer electrode in the surrounding atmosphere when high voltage (kV) pulses with nanosecond duration and kHz repetition rate are applied to the electrodes. The use of this plasma source type focuses on biological applications of plasmas [14,15].

The use of radio frequency (RF) voltage supplies favors the operation of plasma sources. The breakdown of a discharge is frequency-dependent, showing a minimum in the RF domain [16] which is valid for a large range of pressures. This means that in comparison to DC discharges, the plasma will be not only easier to ignite, but will operate at lower current and power, presenting for the same pressure range, less risk for instabilities. Consequently, plasma sources based on the RF discharges are frequently encountered in research and technology. A large variety of discharge geometries (planar, needle, multi-electrode assembly, cylindrical geometry) were designed for specific applications, among them geometries leading to expanding atmospheric-pressure RF plasmas. The common expanding plasma sources based on RF discharges may be assigned to the following categories:

(i) *Hollow cathode expanding plasma jet systems*. In these systems, the RF power is applied to a hollow electrode, usually a metallic tubing, which may or may not contain another dielectric tubing inside. The gas admission is done through the inside of the electrode, and the jet emerges from the inside of the RF electrode. In the low-pressure sources, the RF electrode is inserted in the vacuum chamber, or in the case of atmospheric pressure sources it penetrates in open atmosphere. Various groups reported the development and use of such "hollow cathode" systems [17–22]. Concerning their use, the research pointed to deposition. The process was based either on material sputtering or evaporation from the inside of the cathode, either on plasma-assisted chemical vapor deposition (PACVD), with downstream precursor injection. Hollow cathode jet sources were used for deposition of nitrides [23], oxides, or even diamond [24]. In order to fulfil requirements for deposition of composite films, such sources were assembled in multi-jet systems [25] or, for large area processing, as fused hollow cathode devices [26].

(ii) "*Plasma needle" systems*. This system is based on a thin wire electrode enclosed by an insulator glass tubing of small diameter, which ends in open atmosphere [27,28]. The gas used is mainly helium. The tubing has the role of directing the gas flow around the electrode, protecting it against damage, and carrying out the excitation outside the discharge. The device is powered by small RF voltages at 13.56 MHz and provides a small plasma attached to the wire end. This plasma is intensively studied in relationship with biological and medical applications [29,30].

(iii) *Atmospheric-pressure plasma jet (APPJ) systems*. They make use of discharges generated in narrow spaces, without any barrier in-between. In its cylindrical version [31], the APPJ consists of two coaxial electrodes separated by a small space. The gas used is helium, or helium with a small amount of other gases. The gas is ionized in the annular space delimited by the two electrodes. The outer electrode is grounded and narrowed at the end where the plasma exits, playing the role of nozzle. In its rectangular version, the APPJ [32] consists of a rectangular channel delimited of two parallel-plate electrodes, and two insulating spacers which keep the plates separated by a few millimeters distance. The gas is introduced in the channel at one end, is homogeneously excited in the gap, and exits the other end as a two-dimensional cold plasma beam. In a third variant, the two electrodes are parallel but perforated (sieve electrodes) and the helium gas passes perpendicularly on them, producing a volumic expanding plasma with a larger cross-section. Such systems were extensively searched for various applications, e.g., deposition [33] and etching [34].

The common difficulties associated with the above-mentioned expanding RF plasmas are the tendency to arcing and filamentation with the pressure increase. The cause of this tendency stands in the thermal instabilities driven by the gas and electrode heating [35]. Consequently, solutions for efficient cooling were considered, the most frequent being the use of cooled electrodes, of high gas flows, and, if possible, of gases with high thermal conductivity (helium, for example).

Herein we report on the design and characteristics of an RF expanding plasma source jet (RF-jet) based on a closed-volume, parallel-plate discharge configuration, with the plasma ejecting through a nozzle performed in the grounded electrode. The discharge does not make use of dielectric barriers and can be operated at low, intermediate, and atmospheric pressure in various gases including argon, nitrogen, and air. The discharge phenomenology associated with the extension of plasma sources operation from low to atmospheric pressure shows that cold plasmas can be obtained. Specific applications of this kind of plasma sources are presented, amongst them the growth of carbon nanowalls, cleaning of surfaces from carbon residues, and the patterning of polymeric foils with wettable traces.

SOURCE OPERATION

Discharge configuration of RF-jet sources

The discharge configuration (Fig. 1a) consists of a parallel-plate geometry, in which the two disk-shaped electrodes are separated by a small gap (D) of 1–5 mm. The upper electrode is RF-powered, while the lower one is grounded. A hole (aperture, $d = 0.5$–3 mm) machined in the grounded electrode plays the role of nozzle. An insulating (ceramic, quartz) cylindrical tube closes around the discharge space and defines the discharge chamber of the plasma source. The feeding gas (argon, nitrogen) flows through the discharge chamber, passes across the gap, and expands through the nozzle outside the inter-electrodic space.

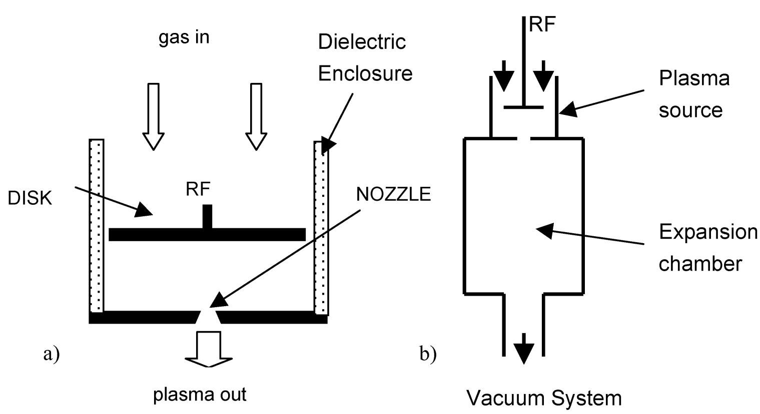

Fig. 1 (a) Discharge configuration. (b) Set-up configuration for plasma source operation at low pressure.

Low-pressure operation

For low-pressure operation, the plasma source is mounted on an expansion chamber (Fig. 1b) which is pumped out by a vacuum system. The discharge is mainly sustained in the narrow gap defined by the powered RF electrode and the planar nozzle, but can be transferred through the aperture in the grounded expansion chamber. The conditions allowing the transferring of discharge in the absence of the gas flow have been discussed previously [36]. The transferring takes place only if the development of the sheath is allowed inside the hole. The sheath thickness is a decreasing function of the discharge power and pressure. Starting with low values of these parameters and a given hole radius, the transfer condition is fulfilled by increasing power or pressure. Then, plasma expands in the expansion chamber. If the start values of pressure and power are already compatible with the transferred discharge, then the breakdown occurs simultaneously in the interelectrodic gap and in expansion.

In presence of the gas flow the produced plasma is directional due to the transport phenomena. At low pressure, the collisional processes leading to recombination and loss of excitation have low rates and plasma extends on large distances (many tens of centimeters). An image of a nitrogen plasma beam generated at low pressure by an expanding plasma source is shown in Fig. 2.

Fig. 2 Image of a low-pressure nitrogen plasma beam (100 W, 2 mbar).

Phenomena associated with the extension of plasma source operation at high pressure

Investigation of the discharge operation during pressure increase by image recording

The image recording was performed for the interelectrodic space (through the insulating quartz tube) and for the expanding region (realized in a glass tube connected to a vacuum system, as in Fig. 2) in argon and nitrogen. The RF electrode has been provided with active water cooling. Large enough gas mass flow rates (up to 10 000 sccm) and power values (up to 300 W) were used in order to maintain the operation up to atmospheric pressure. The experimental procedure consisted of breakdown at low pressure (10^{-2}–10^{-1} mbar), followed by the gradual pressure increase at constant mass flow.

Various discharge stages are encountered with the pressure increase, peculiar to the two gases. In argon, at low pressure (~10^{-1} mbar), the discharge fills uniformly the inside gap volume and extends through the nozzle outside the interelectrodic space. The pressure increase leads to discharge constriction, and a narrow plasma column fixed on the nozzle exit and in the opposing point on the upper electrode occurs. This discharge constriction appears to be a gradual, continuous process. The coexistence of diffused and constricted discharge is observed in the gap at intermediate pressures. The expansion of the column is the origin of the plasma jet at high pressures (Fig. 3), including the atmospheric one. The passage of the plasma beam from low to atmospheric pressure is smooth, without instabilities or transitions in the discharge regime.

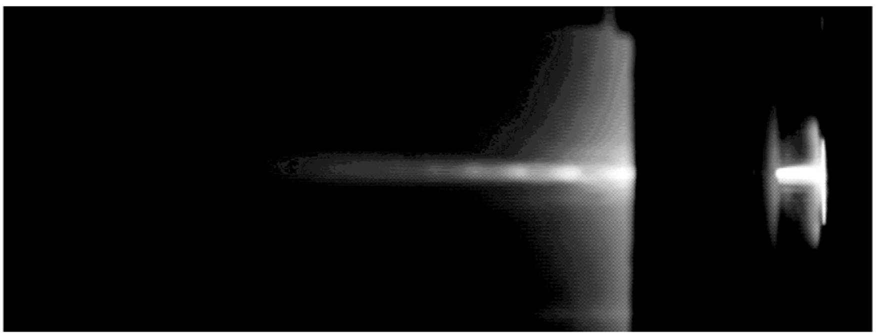

Fig. 3 Image illustrating that the origin of the expanded plasma beam is the constricted plasma column (argon, 50 W, 1015 mbar).

In nitrogen, the pressure increase leads as well in a continuous manner to the columnar discharge, but the coexistence of diffuse and constricted discharge is doubtful. Moreover, the constricted plasma is instable, the columnar discharge channel running around inside the narrow interelectrodic space, while the beam in expansion tends to extinguish. Nevertheless, at some pressure value (depending on geometry, in the range 200–500 mbar) the plasma column anchors on the nozzle exit, and in the opposite point on the RF electrode, like in the argon case. Further increase of pressure leads to a stable regime, characterized by a stable jet even at over atmospheric conditions. Apparently, in the nitrogen case the passage of the system from low to atmospheric pressure is affected by instabilities, which cease at the moment of column anchoring on the nozzle exit.

The very important characteristic, that the plasma jet at high pressures, including the atmospheric one, is sustained by the constricted plasma column (Fig. 3), which has low radial dimension, allows the design and the construction of small-diameter plasma sources, as will be presented further in the Section "Practical examples of RF-Jet plasma sources and their applications".

Electrical characteristics

The electrical measurements were performed by using a Tektronix digital oscilloscope (type 2432A), with current and voltage probes (type P6021 and P6137) via an interface connected to a computer. A program conceived in the Labview environment allowed the gradual increase and decrease of the forwarded power, and recording of current and voltage values.

One of the I–V characteristics, obtained for the operation of the plasma beam discharge at 0.5 mbar and 2000 sccm, is presented in Fig. 4a. The A to B portion of the curve describes the behavior up to breakdown. The linear increase of current with voltage, even in plasma absence, is explained by the capacitance of the gap. After breakdown the discharge parameters jump to point C. The behavior of the discharge for further increase of power is described by the portion C to D. The increase of the current upon voltage in this portion suggests an abnormal glow regime. From D, where the power turns to decrease, the discharge passes through the extinction point E and the cycle is closed by the not-existing discharge portion, from E to A.

A curve similar to that from Fig. 4a is presented in Fig. 4b, for a discharge operated at 1000 mbar and 2000 sccm.

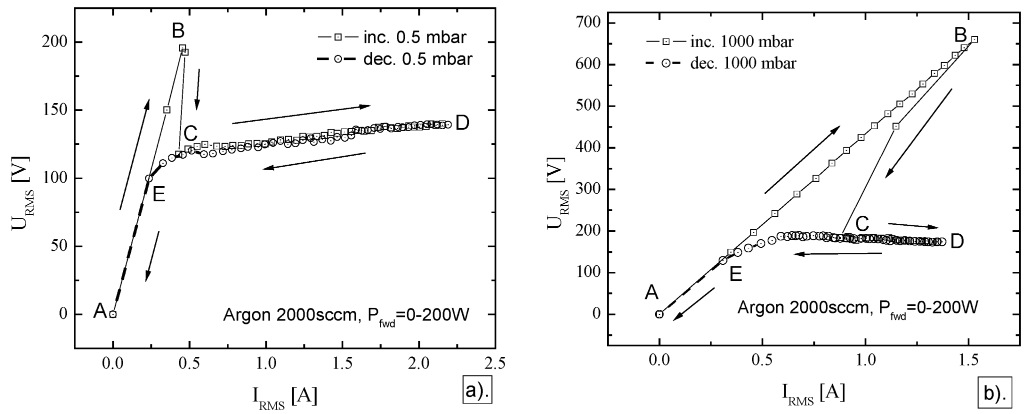

Fig. 4 (a) I–V characteristic of the plasma beam discharge in argon, at 0.5 mbar and 2000 sccm, and (b) I–V characteristic of the plasma beam discharge in argon, atmospheric pressure, and 2000 sccm. (The points correspond to a steep increase/decrease of the RF forwarded power with 5 W in the power range of 0–200 W).

Nature of the plasma beam discharge

The equilibrium or nonequilibrium nature of the constricted plasma column from which the expansion occurs, and of the plasma beam itself, is a very important issue, related to the use of the plasma source in specific applications.

The absence of change in plasma characteristics from the low to intermediate and atmospheric pressure range suggests that the plasma remains in a nonequilibrium state until the atmospheric pressure is reached. Spectral investigation of the beam at atmospheric pressure with cooled electrodes does not show emission of the electrode material lines, in contrast with the case when not active cooling is performed. In the absence of cooling, a strong transition in the plasma state appears, with bright light and strong emission of electrode material atomic lines, corresponding to the onset of an RF arc. In accord with these elements, we have reasons to consider that the configuration described, in the presence of high mass flows and cooled electrodes and at reasonable power levels, produces cold plasma at atmospheric pressure. The relatively low gas temperatures measured by a thermocouple inserted in the expanded jets, presented further in this paper, for various gas flows and power levels, sustain as well the cold plasma hypothesis. The situation could be probably parallelized to the nonequilibrium stage of the gliding arc discharge [37], except that now plasma is stationary.

Comparison with other expanding plasma sources

In comparison with the "hollow cathode" systems, the RF-jet generated by the discharge configuration described in Fig. 1a does not originate in any hollow part of the RF electrode, but in the plasma volume developed outside a flat RF electrode. There are substantial constructive and functional differences between the presented plasma source and the "plasma needle" designs. This source does not use a wire as electrode, is easily operated in other gases than helium, uses a counter electrode as nozzle, can work at low and large power values, and at low and atmospheric pressure. In this source, an external dielectric enclosure closing laterally the discharge region is used, impeding the discharge development in the annular space between the electrode holder and the grounded external jacket. Consequently, contrary to the APPJ systems, the RF-jet formation is not based on the expansion of homogeneous volume plasmas, but it originates in the constricted plasma column formed at high pressure, and not necessarily in helium, but in argon, nitrogen, and even air. Both the design and the operation principle are very different in comparison to those used in "plasma pencil" systems, which, in addition, works with high voltage, hundreds of nanosecond pulses. In comparison to the hollow anode (or constricted plasma source variant) sources operated with DC or bipolar pulsed voltages at low pressure, this RF plasma source based on flat electrodes is functional in the pressure range from 10^{-3} mbar to over-atmospheric. By their characteristics, the presented plasma sources are completing the range of the other expanding sources, finding a place for improvment or opening new ways for applications.

PRACTICAL EXAMPLES OF RF-JET PLASMA SOURCES AND THEIR APPLICATIONS

Low-pressure growth of nanostructured materials by low-pressure RF expanded plasma

At low pressure, the expanding RF plasma sources (RF-jets) are suitable for PACVD, e.g., for the growth of nanostructured carbon layers [38,39], or for increasing the reactivity during pulsed laser deposition of functional oxide thin films [40]. The experimental set-up used for the growth of carbon materials with various morphologies is presented in Fig. 5a. The carbon species sustaining the growth are supplied by the expanding plasma beam generated in argon, and injected downstream the nozzle with low amounts of acetylene and hydrogen. The typical process parameters were RF power 250 W, pressure 10^{-2}–3 mbar, argon mass flow rate 500–2000 sccm. The corresponding plasma source (60 mm diameter) is shown in Fig. 5b, where there are visible details like the exit nozzle, and the route for acetylene introduction ending in the injection ring. The growth occurs on oxidized silicon substrate heated at ~700 °C. The catalyst sustaining the growth is deposited by sputtering of a Ni target with a small-size magnetron mounted laterally on the expansion chamber. The capability of the source to lead to combined growth of nanotubes and nanowalls (as shown in Fig. 5c) was demonstrated recently [41].

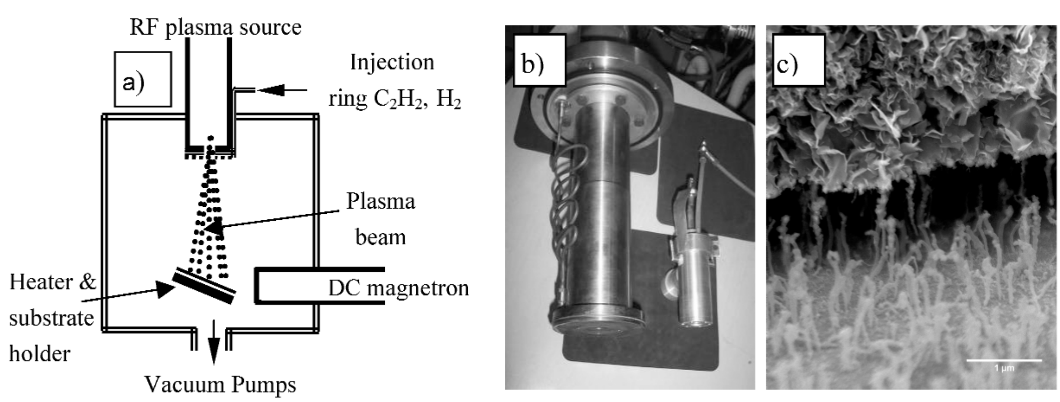

Fig. 5 (a) The experimental set-up used for the growth of carbon materials. (b) Image of the plasma source (source diameter 60 mm). (c) SEM image of carbon nanotubes and carbon nanowalls obtained in one-step combined growth.

Carbon material removal from surfaces by a small-size plasma torch

This application was developed in relation with cleaning of the carbon layers which are co-deposited with tritium on the castellated tiles of fusion machines [42]. The requirements for the source suitable for this application were small size for allowing access in hidden and narrow spaces, stability, and easy handling, capabilities to affect the surface by physical and chemical processes, flexibility for mounting on robotic arms. The realized RF-jet plasma source is presented in Fig. 6a. The source is manufactured in stainless steel, has 20 mm diameter, and uses refractory materials inside. Both the internal electrode and the external jacket are provided with active water cooling. The source works under stable conditions both in argon and nitrogen. Operation with powers up to 600 W was possible by means of adequate cooling. The breakdown of the discharge sustaining the jet can be realized directly at atmospheric pressure. The temperatures attained by a thermocouple inserted in the plasma beam, at various distances from the nozzle, are presented in Fig. 6b. Temperatures of 1000–1200 °C are noticed in nitrogen in the nozzle vicinity, and in the range 800–900 °C at 8–10 mm from the tip nozzle, almost twice the values in argon. Surface experiments were realized with the nitrogen plasma scanning of graphite and carbon fiber composite (CFC) specimens. In the case of graphite roughening of surface, and material removal was measured by profilometry. The experiments with CFC tiles from Tore Supra Tokamak indicated material removal with a rate of 2.5 10^{-4} g/min. The material removal is done by a chemical process and does not lead to dust formation.

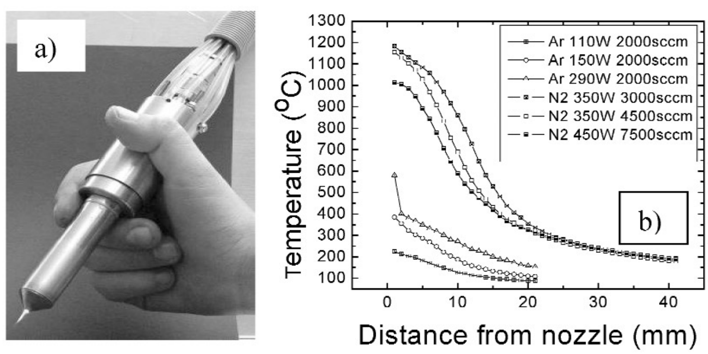

Fig. 6 (a) Hand-held flexible 20-mm-diameter plasma source. (b) Dependence of temperature upon position at various powers and mass flow rates of argon and nitrogen.

Patterning of polymeric foils with wettable traces

The previous plasma source is not suitable for treatment of temperature-sensitive substrates (polymers, biological samples) due to the thermal loading of substrates. In view of such applications, a smaller-size, lower power plasma source was realized on the same principle (Figs. 7a and 7c). The source has only 8 mm diameter, is not provided with cooling, and can be operated in argon with powers in the 15–25 W range at mass flow rates of 2000–4000 sccm. The plasma jet diameter is 0.5–1 mm, and its length is 1–10 mm, depending on the gas, mass flow rate, and power. The temperatures attained by a thermocouple inserted in the beam are in the range 40–100 °C (Fig. 7b). The source is suitable for scanning procedures of polymeric foils as shown in Fig. 7c. The change of contact angle with the number of scans is presented in Fig. 8a. Even after the first scan (scanning speed 5 mm/s) the contact angle decreases from 75 to 42 °C and a stationary value of 34 °C is attained after 4 scans. In addition, the effectiveness of the source in promoting the wettability of polyethylene terephthalate (PET) foils was demonstrated by condensing water vapors from the ambient atmosphere on cooled neighbor regions of treated and nontreated surfaces. The cooling was realized by placement of the foil on the surface of a Peltier element (Fig. 8b). The different shapes of the water drops, on the treated and nontreated regions,

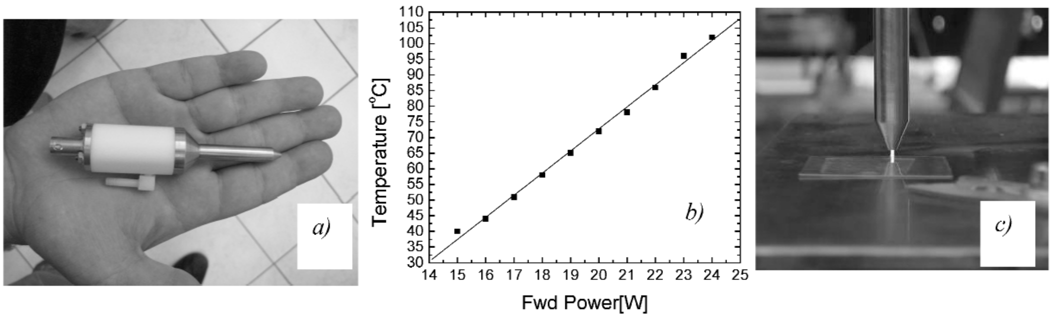

Fig. 7 (a) Low-power, small-size atmospheric-pressure plasma source for processing of temperature-sensitive substrates (source diameter: ~8 mm). (b) Dependence of the temperature on the forwarded power. (c) Small-size plasma source during treatment of a polymeric foil (scanning speed 5 mm/s).

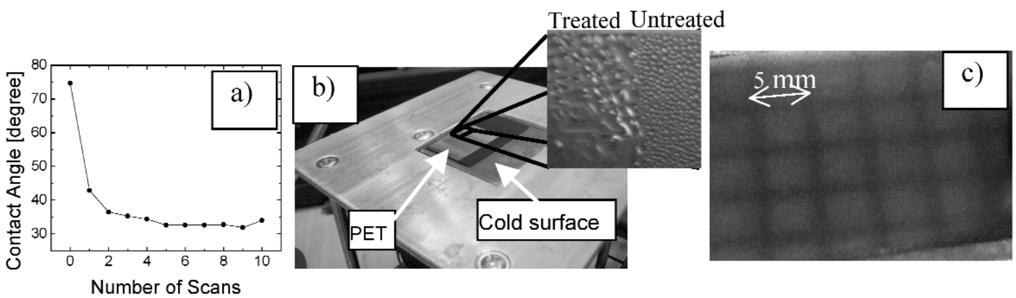

Fig. 8 (a) Dependence of the water contact angle upon the number of scans. (b) Illustration of surface modification, by condensation of water vapors on cooled, treated, and untreated surfaces. (c) Illustration of surface patterning with wettable traces, by condensation of water vapors on cooled PET surface.

give different appearances of the treated and untreated zones in photographs. Moreover, an experiment has been performed in which the PET surfaces were scanned on perpendicular tracks separated by 5 mm distance. The image of the foil treated in this manner, placed on a cooling Peltier element, is shown in Fig. 8c. It demonstrates that the polymeric foil has been patterned with wettable traces (width about 1 mm), as proved by the visible different appearances of the treated and untreated areas.

Other applications

There is an increasing interest in application of the atmospheric-pressure plasma sources for the treatment of biological samples and liquids processing. By handling the source parameters, cold plasmas are produced. The surrounding of the discharge with a grounded jacket in which a hole is performed is one of the advantages of the RF-jet system: (i) the electromagnetic field is screened and (ii) the RF electrode and the discharge generating the jet are not accessible from outside, so the plasma source is able to come in contact with the human tissues without voltage danger and generation of burning effects. That might be of interest for medical applications. The ability of expanding RF sources to work at over-atmospheric pressure allows their operation under water (as shown in Fig. 9), opening conditions for depollution applications or plasma chemistry processing in liquid conditions.

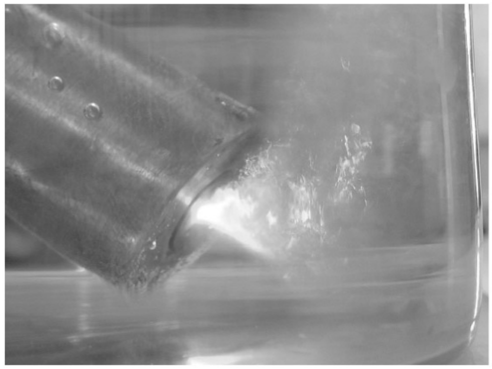

Fig. 9 Underwater operation of over-atmospheric-pressure RF-jet.

CONCLUSIONS

The expansion of RF discharges outside closed-space, small inter-electrode gaps opens the way for designing RF plasma sources (RF-jets) with continuous operation from low to atmospheric and even over-atmospheric pressure. The sources operate with bare electrodes. Direct breakdown at atmospheric pressure was proved. The expansion of the constricted plasma column is at the origin of expanded plasma beam in the case of intermediate and atmospheric pressures. The small radial extension of the constricted plasma column allows the construction of small-size plasma devices. Sources of 60, 20, and 8 mm diameter are presented in the paper. The stable operation in argon, nitrogen, and pure air was demonstrated with various RF-jet sources, for a large range of power values (from 600 to 15 W) and mass gas flow rates (100–10 000 sccm). At low pressure, the plasma beams generated by the expanding RF discharge proved their advantages in assisting other deposition techniques, e.g., pulsed laser deposition, or as direct plasma environment for PACVD processing, including the growth of carbon nanotubes and nanowalls. At atmospheric pressure, the sources proved to be effective in the cleaning of residual carbon from surfaces, being prospective tools for removal of co-deposited layers and detritiation of Tokamak tiles. The atmospheric- pressure, small-size (8 mm), small power (15–25 W) plasma source has a low thermal loading effect and allow the treatment of temperature-sensitive materials. The patterning of PET surfaces with wettable traces was presented as an example. The sources are suitable for medical applications being able to come in contact with the human tissues without voltage danger and generation of burning effects. Due to their ability to be operated in underwater conditions, the RF-jet plasma sources are promising tools for plasma chemistry processing in liquid media.

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