Atomic Oxygen Maximization in High-Voltage Pulsed Cold Atmospheric Plasma Jets

Nicolae Georgescu, Cristian P. Lungu, Andreea Roxana Lupu, and Mariana Osiac

***Abstract*—This paper presents a new device generating high-voltage pulsed cold atmospheric plasma jets. With these plasmas, the quantity of atomic oxygen (and, accordingly, the chemical activity) is a lot higher than that in previous researches. The main characteristic of the new device is the usage of three tubular needle-type electrodes connected in parallel. By applying high-voltage pulses (with 20–30-kV amplitude, duration of hundreds of nanoseconds, and hundreds of pulses per second), three independent discharges are formed in the discharge room. The plasma-forming gas is He, with a low quantity of oxygen introduced through the high-voltage electrodes. Each discharge has an optimal percentage of 0.5% vol. O_2 in He, which maximizes the quantity of atomic oxygen in the plasma. The plasmas of the three discharges unite to a single jet though, which contains a quantity of atomic oxygen that is a lot higher than that of the plasma of each discharge. The emission spectra of the plasma jets show the maximization of the intensity of the O I 777 nm line when a concentration of 1.5% vol. O_2 in He (three times higher than so far) is introduced in the plasma-forming gas.**

***Index Terms*—Atomic oxygen, cold atmospheric plasma jets, pulsed high voltage.**

I. Introduction

ATMOSPHERIC pressure cold plasmas are still intensively studied for applications such as modifying the properties of certain surfaces [1]–[3], physical and chemical modifications of polymers [4], thin-film deposition [5]–[7], nanotechnologies [8], and depollution of gaseous or liquid environments [9], [10]. However, the largest part of applications of these plasmas belongs to the biomedical field: microorganism destruction (decontamination of biological, food-related, etc., media) [11]–[14], *in vitro* and *in vivo* cell treatment [15]–[20], and various medical treatment tests [21]–[25]. All these applications belong to one of the newest research fields of the last few years: plasma medicine [26], [27]. This paper regards plasma medicine as well: We aim to increase the efficiency of the atmospheric cold plasma jets treatment of microorganisms and of normal and tumoral cells.

There are different types of atmospheric pressure cold plasma sources, depending on the voltage applied between the electrodes of the device employed: microwave voltage, radio-frequency voltage, ac voltage (tens of kilohertz), and high pulsed voltage. An inert gas (He or Ar) is the main plasma-forming gas. For biomedical applications, it is, however, necessary for the plasma to be chemically activated. That is why minute amounts of an additional gas as the source of reactive species are used. In most cases, reactive plasma is generated as a jet, which allows for the rapid transport of the excited species on the treated object.

In this paper, just like in our previous ones [28]–[30], the interelectrode voltage applied for obtaining the plasma is pulsed high voltage, with amplitudes of 20–30 kV, 100–500-ns width at half maximum, and frequencies of tens of hundreds of pulses per second (pps).

Molecular oxygen is used as the source of reactive species in our research.

The chemical processes start with a generation of atomic oxygen, produced by electron impact dissociation [31], by dissociative attachment [31], and by Penning ionization [32].

Little is known about the way in which atomic oxygen acts on bacteria and animal cells. It was suggested [33] that atomic oxygen may be responsible for the oxidation of proteins. The efficiency of the atomic oxygen in destroying species with high clinical significance has been shown for *Escherichia coli*, *Pseudomonas aeruginosa*, and *Bacillus cereus* [34]. The atomic oxygen plays an important role in the inactivation of many other bacterial strains, proving its applicability in the sterilization of culture media, material surfaces, and air [26], [33], [35], [36]. This makes it ideal for cleaning surgical implants, which are often contaminated with fragments of bacteria cells even after sterilization.

Nevertheless, by the reactive oxygen species that it produces, the atomic oxygen is indirectly involved in the destruction of pathogens and induction of apoptosis or necrosis in mammalian cells, including cancer ones [26], [35].

In order to obtain higher quantities of atomic oxygen, it is necessary to increase the concentration of molecular oxygen (O_2) in plasma-forming gas (He, in our case). Unfortunately, this increase brings along the decrease of the plasma current. Consequently, the quantity of atomic oxygen produced within the plasma has an initial increase along with the concentration of molecular oxygen, and then, it drops as a result of the decrease of the plasma jet current. Therefore, there is an optimal concentration of O_2 in He/Ar that maximizes the quantity of atomic oxygen.

For the RF voltage supply of the plasma jet, experimental works [37] have determined an optimal concentration of 0.5%–0.6% vol. O_2/He. Using a model that considers

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N. Georgescu, C. P. Lungu, and A. R. Lupu are with the National Institute for Laser, Plasma and Radiation Physics, 077125 Bucharest, Romania (e-mail: ngeorge@infim.ro; cristian.lungu@inflpr.ro; ldreea@yahoo.com).

M. Osiac is with the University of Craiova, 200585 Craiova, Romania (e-mail: m_osiac@yahoo.com).

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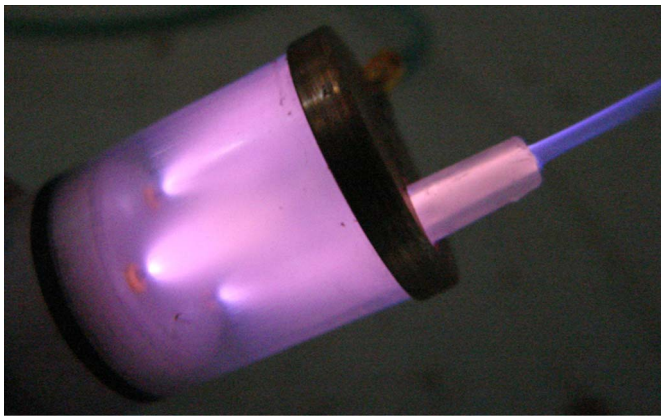

Fig. 1. High-voltage pulsed cold atmospheric plasma jet generator (the initial transparent device).

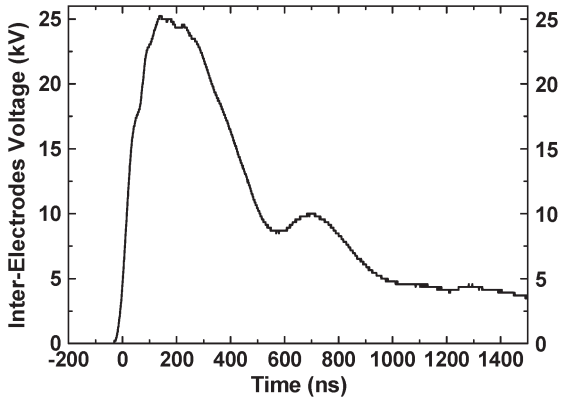

Fig. 2. Voltage applied between the high-voltage electrodes (syringe needles) and the ground electrode.

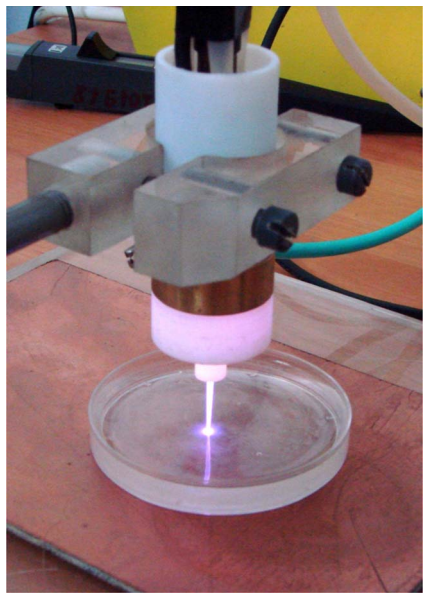

Fig. 3. Final device, generating high-voltage pulsed cold atmospheric plasma jet.

35 dominant chemical reactions in the RF He/O_2 plasma, an optimal theoretical value of 0.5% vol. O_2/He [38] has been obtained. Regarding the submicrosecond pulsed atmospheric plasma (our case), the value considered as optimal for the oxygen concentration is 0.5% vol. O_2/He [39] as well.

In all the papers mentioned earlier, a coaxial device with high-voltage central electrode was used for generating the plasma jet. The ground electrode was mounted on the exterior, separated from the central electrode by a dielectric barrier.

In the following paper parts, we will describe a new pulsed cold atmospheric plasma jet-generating device, which allows for the increase of the optimal concentration of molecular oxygen in He and, consequently, for the increase of the quantity of atomic oxygen in the plasma jet. The experimental results will be presented and commented as well.

II. Experimental System

The key component of the experimental system is the high-voltage pulsed cold atmospheric plasma jet generator (Fig. 1). A dielectric piston is placed inside a cylinder (inner diameter = 20 mm), which is also a dielectric. The piston is penetrated by three medical syringe needles. On the surface of the piston, the exit orifices of the needles are evenly placed, on a circle with a radius of 8.5 mm. The part of the needle coming out of the piston is 1.5 mm long. The opposite ends of the needles are electrically connected in parallel. In addition, oxygen is introduced through the needles, as a chemical activation gas. The inert gas (He) is introduced through the center of the piston. The syringe needles behave as high-voltage electrodes. The ground electrode is the opaque copper part, mounted at the end of the dielectric cylinder, around the exit nozzle. Electrical discharges are produced in the mixture of He/O_2 (the oxygen concentration being of a few percentages only) by applying high-voltage pulses in the shape shown in Fig. 2 between the syringe needles and the ground electrode. The space distribution of the syringe needles allows for three independent electrical discharges to be produced inside the discharge room (Fig. 1). The plasma of the three electrical discharges is pushed toward the gas flow through the exit orifice, taking the shape of a unique plasma jet.

The final variant of the device (Fig. 3) is made of Teflon, for increasing the reliability when working with high-voltage pulses. In this case, the ground electrode surrounds the Teflon cylinder at 1-cm distance from its end.

For the cell treatment, the cells are immersed in culture media. To simulate this type of experiments, the plasma jet falls down on the surface of a tap water (Fig. 3). This water has an electrical conductivity of 800–1000 $\mu S \cdot cm^{-1}$, in the range of electrical conductivities of cell culture media. The tap water container, with a diameter of 55 mm, is placed on a copper plate electrically connected to the ground.

The voltage/current pulses are measured with Tektronix P6015A and P6012 probes, respectively. The probes are coupled to the Tektronix TDS 2024B oscilloscope.

The gas flow rates have been measured by using flow controllers: 1) Cole Parmer type with a range of 0–5 L/min (for He) and 2) Aalborg GFC-17 type with a range of 0–50 mL/min (for O_2).

The emission spectra of the plasma jets were obtained using an Ocean Optics HR4000 spectrometer. Its optical resolution is 0.75 nm full width at half maximum. The spectra are displayed

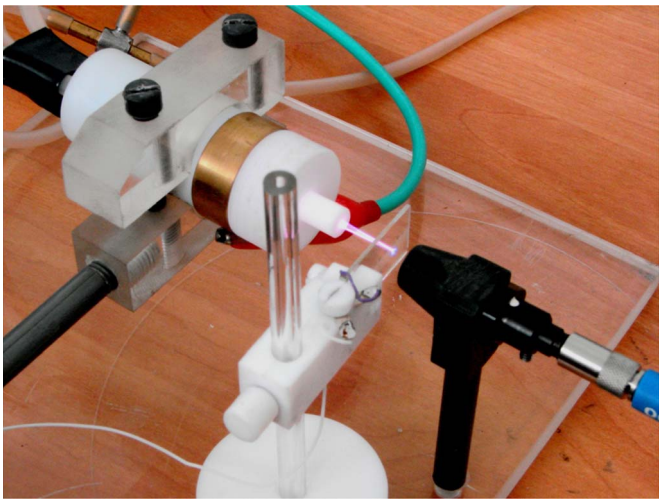

Fig. 4. Arrangement which allows that the spectrometer observation axis be collinear with the plasma jet (axial view).

on the computer screen, using SpectraSuite. The spectrometer allows for the adjustment of the integration times so that the saturation problems should all be solved.

For result confidence, the plasma emission spectra have been noticed on two directions.

1) The observation axis is perpendicular to the plasma jet (side view). The lens of the spectrometer aimed at an area that was 2 mm away from the exit orifice of the jet.
2) The observation axis is collinear with the plasma jet (axial view). In this case (Fig. 4), the plasma jet hits a glass plate on the side where a thin (200-nm) layer of indium tin oxide (ITO) was deposited by magnetron sputtering. This layer is optically transparent and has a good electrical conductivity. A metal wire makes the electrical contact between the layer and the water in the dielectric container. The lens and the optical fiber making the connection with the HR4000 spectrometer "look" from the other side of the glass plate, along the axis of the plasma jet.

III. Experimental Results and Discussions

During our previous research, plasma jets have been generated using cylindrical devices, with a unique high-voltage electrode axially placed in the device [30]. In these conditions, we have experimentally demonstrated that the optimal concentration of O_2 in He that maximizes the production of atomic oxygen was of 0.5%, congruent with the results of other authors [37]–[39].

In this paper, in order to determine the optimal working voltage, we initially monitored the evolution of the O I 777 nm line (the unresolved triplet O I 777.194, O I 777.417, and O I 777.539 nm) intensity depending on the amplitude of the interelectrode high-voltage pulses. This spectral line corresponds to $3\ ^5\mathrm{P} - 3\ ^5\mathrm{S}^0$ atomic oxygen transition, according to the U.S. National Institute of Standards and Technology database. Fig. 5 shows the dependence of the O I 777 nm line intensity of the interelectrode voltage amplitude, for two O_2 admixtures of 1% and 1.5% in the discharge. The O I 777 nm line intensity is rising linearly with discharge voltage. The increase in this line

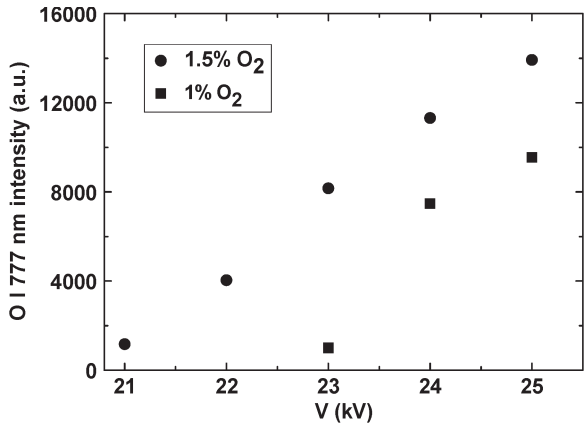

Fig. 5. Dependence of the O I 777 nm line intensity of the interelectrode voltage amplitude, for two O_2 admixtures in He: 1% and 1.5%. He flow rate: 2.5 L/min. Pulse repetition frequency: 100 pps. Spectrometer integration time: 600 ms. Side view.

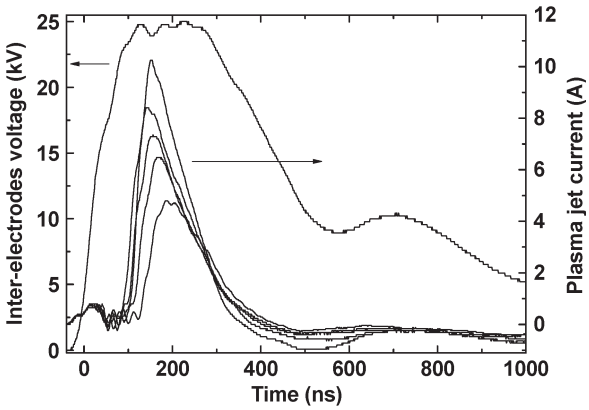

Fig. 6. Plasma jet currents at several O_2 concentrations, for the same interelectrode voltage pulse. From the most intense to the weakest current, the O_2 concentrations are 0%, 0.5%, 1%, 1.5%, and 2%, respectively. He flow rate: 2.5 L/min.

intensity could be explained by assuming an increasing electron energy with the input voltage, which increases the number of O_2 dissociations. Under these circumstances, the subsequent experiments were performed with 25-kV voltage amplitudes.

Fig. 6 shows the current pulses at several concentrations of O_2 in He, for the same interelectrode voltage pulse. We notice the constant decrease of the plasma jet current together with the increase of the concentration of molecular oxygen. The quantity of atomic oxygen, highlighted by the O I 777 nm line, shows, however, a different evolution.

Fig. 7 shows plasma jet emission spectra for several concentrations of oxygen in He. The observation axis is perpendicular to the plasma jet (side view). The He flow rate is 2.5 L/min. The voltage pulses between the electrodes have the shape shown in Fig. 2 and are repeated with a frequency of 100 pps. All emission spectra reveal the presence of excited He and atomic oxygen in the plasma jet, as well as some excited N_2 molecules (from the air entrained in the plasma jet). The He (587 and 706 nm) and N_2 (337–406 nm) lines have maximum amplitudes at zero concentration of O_2 in He (Fig. 8). The addition of O_2 to the plasma-forming gas led to significant decreases of the He and N_2 lines, but without affecting the atomic oxygen emission

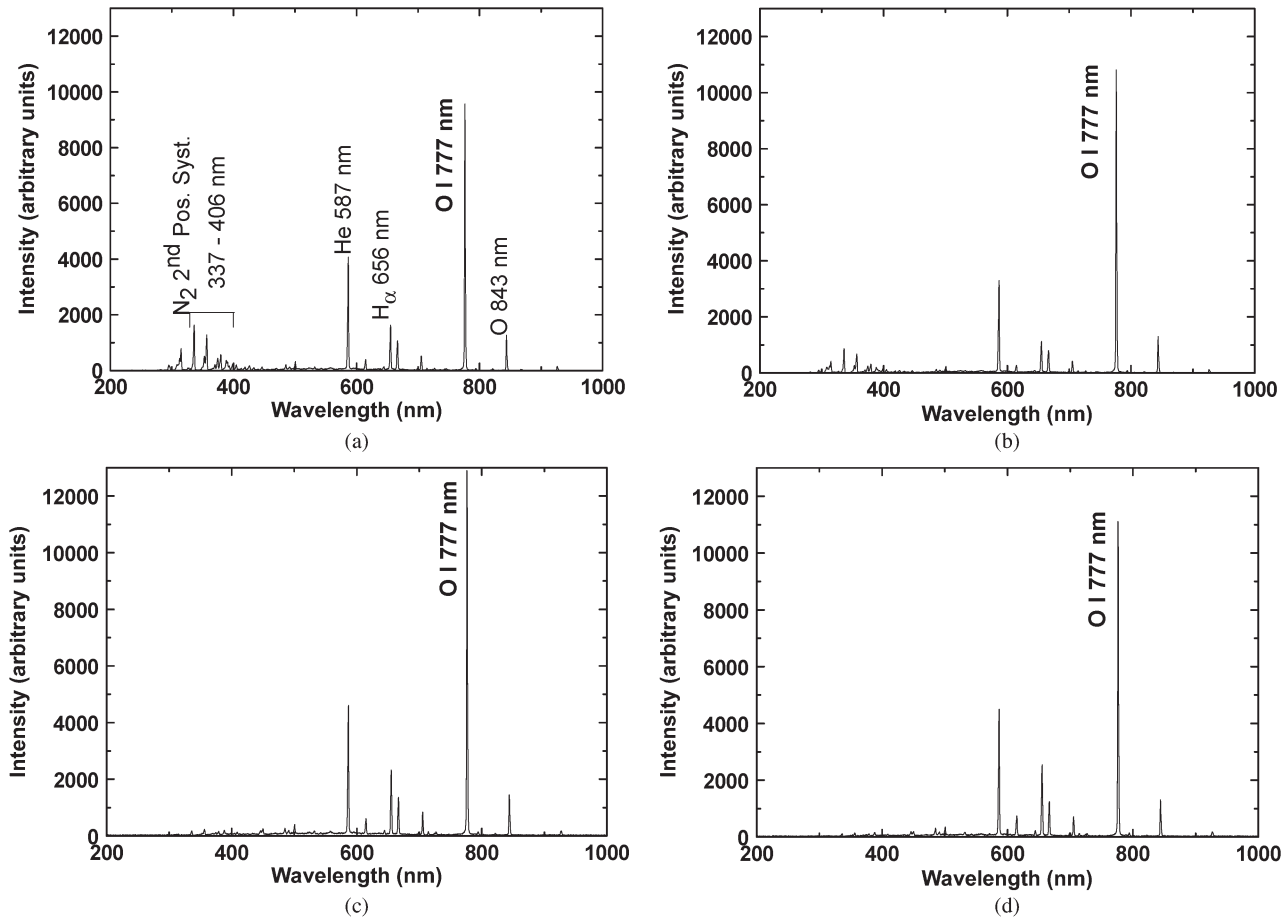

Fig. 7. Plasma jet emission spectra for several concentrations of oxygen in He: (a) 0.5%, (b) 1%, (c) 1.5%, and (d) 2%. The observation axis is perpendicular to the plasma jet (side view). He flow rate: 2.5 L/min. High-voltage amplitude: 25 kV. Pulse repetition frequency: 100 pps. Spectrometer integration time: 600 ms.

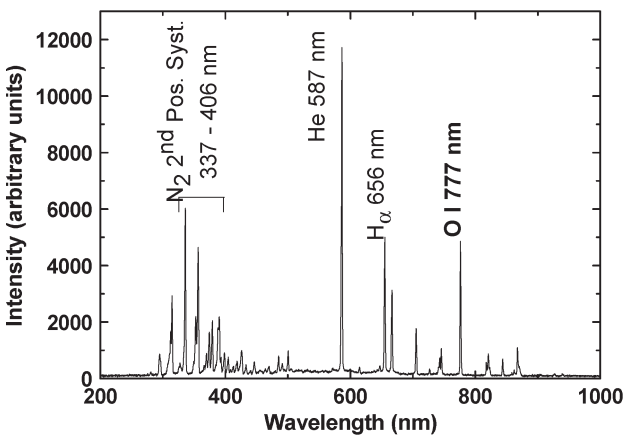

Fig. 8. Main spectral lines of the emission spectra of the plasma jets, with He only as the working gas. He flow rate: 2.5 L/min. High-voltage amplitude: 25 kV. Pulse repetition frequency: 100 pps. Spectrometer integration time: 600 ms. Side view.

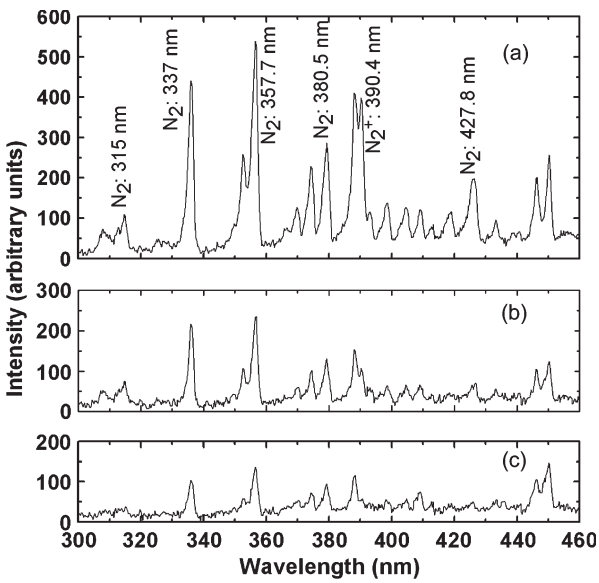

Fig. 9. Plasma jet emission spectra in the 300–460-nm range, for several O_2 admixtures in He: (a) 0%, (b) 0.5%, and (c) 1.5%. He flow rate: 2.5 L/min. High-voltage amplitude: 25 kV. Pulse repetition frequency: 100 pps. Spectrometer integration time: 100 ms. Axial view.

at 777 nm (Fig. 7). This means that a significant fraction of the plasma electrons is used to produce O-containing species from the O_2 molecules, leaving fewer electrons to collide and excite the He atoms and the N_2 molecules.

In Fig. 9, we can notice the decrease of intensity of the N_2 lines together with the increase of concentration of O_2 in He (axial view). With a percentage of 1.5% O_2 in He, the N_2 lines dropped over four times.

As far as the 777 nm line of atomic oxygen is concerned, it has a continuous increase up to a concentration of 1.5% O_2 in He (Fig. 7). We suppose that the O I 777 nm line intensity

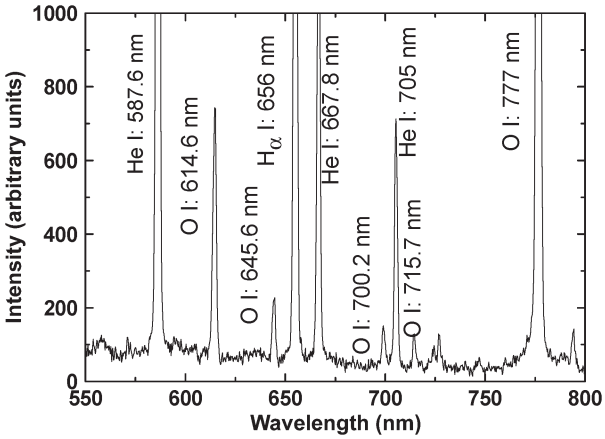

Fig. 10. Occurrence, at 2% O_2 in He, of new spectral lines of atomic oxygen: 614.6, 645.6, 700.2, and 715.7 nm. He flow rate: 2.5 L/min. High-voltage amplitude: 25 kV. Pulse repetition frequency: 100 pps. Spectrometer integration time: 600 ms. Side view.

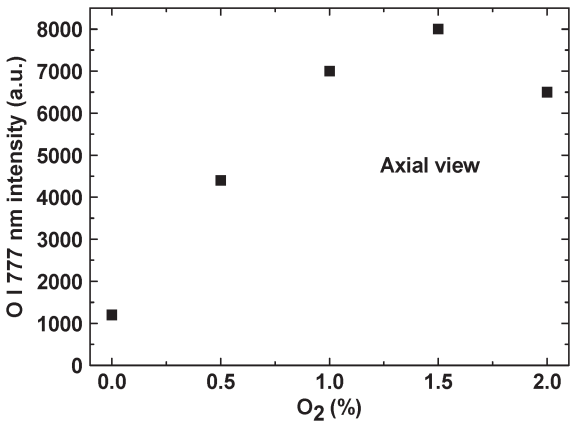

Fig. 11. Dependence of the O I 777 nm line intensity of the O_2 admixtures in He, in the case of axial observation of the emission spectra. He flow rate: 2.5 L/min. High-voltage amplitude: 25 kV. Pulse repetition frequency: 100 pps. Spectrometer integration time: 100 ms.

is related to atomic oxygen concentration. When the value of O_2 in He is over 1.5%, the decrease of the plasma jet current becomes sufficiently important to lead to a decrease of the amplitude of the O I 777 nm line. The interaction between electrons and oxygen molecules also intensifies with high O_2 concentrations. This is demonstrated by the occurrence, at 2% O_2, of new spectral lines of atomic oxygen: 614.6, 645.6, 700.2, and 715.7 nm (Fig. 10).

The same evolution of the O I 777 nm line intensity occurs in the case of axial observation of the emission spectra (Fig. 11). In this case, note that the intensity of the spectral lines is a lot higher compared to that with the side view (the integration time of the spectrometer is only 100 ms, while that with the side view is 600 ms). The explanation is that, with axial observation, the luminous spot (Fig. 4) covers a larger part of the area seen by the lens transmitting the luminous information to the spectrometer.

The presented experiments demonstrate that the optimal value of the concentration of O_2 in He is 1.5%. The obtained value is three times higher than that in the case of experiments in [37] and [39] and of theoretical simulation in [38]. Accordingly, the quantity of atomic oxygen in the plasma jet increases, and the plasma jet becomes more chemically active. The explanation for this increase of chemical activity is that there are three high-voltage electrodes in the discharge room of the new device, which are placed in such a manner that three independent discharges take place. Each discharge has an optimal percentage of 0.5% O_2 in He that maximizes the quantity of atomic oxygen in the plasma. The plasmas of the three discharges unite in a single jet, which contains a quantity of atomic oxygen that is a lot higher than that of the plasma of each discharge.

IV. Conclusion

One method of increasing the chemical activity of the high-voltage pulsed cold atmospheric plasma jets is to form devices with more tubular high-voltage needle-type electrodes electrically connected in parallel. The chemical activation gas is introduced through these electrodes. The electrodes must be placed so that independent electrical discharges occur inside the discharge room. Uniting the plasmas of these discharges in a single jet multiplies the quantity of atomic oxygen in the plasma jet, which becomes more chemically active. The increase of the number of high-voltage electrodes and the appropriate multiplication of the chemical activity of the plasma jet constitute issues to be studied furthermore.

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Nicolae Georgescu was born in Bucharest, Romania, on August 3, 1947. He received the M.Sc. degree from the Polytechnical Institute of Bucharest, Bucharest, in 1970 and the Ph.D. degree in technical physics from the Institute of Atomic Physics, Bucharest, in 1995.

In 1970, he joined the Institute of Atomic Physics as a Physicist Engineer, where he was with the Accelerator Laboratory until 1993. Since 1993, he has been with the Plasma Physics and Nuclear Fusion Laboratory, National Institute for Laser, Plasma and Radiation Physics, Bucharest, as a Senior Scientist. From 1970 to 2000, he worked in the field of pulse power technology (high voltage and high current) with applications for electron accelerators, high-power lasers, and dense magnetized plasmas (plasma focus and vacuum spark). Over the last nine years, he worked for air/water pollution control with pulsed corona systems. Currently, his research interests also include generators of high-voltage pulsed cold atmospheric plasma jets to be applied in the biomedical and the food treatment fields.

Dr. Georgescu is a member of the Romanian Physics Society.

Cristian P. Lungu was born in Bucharest, Romania, on December 16, 1950. He received the M.Sc. degree from the University of Bucharest, Bucharest, in 1974 and the Ph.D. degree in physics from the Institute of Atomic Physics, Bucharest, in 1994.

He is currently the Leader of the Elementary Processes in Plasma and Applications Group, National Institute for Laser, Plasma and Radiation Physics, Bucharest. His main activity is in the field of the plasma processing and applications in waste treatments, plasma displays, and multifunctional film preparations. Using thermionic vacuum arc, an original method developed in his group, more than 1000 inconel and marker tiles were coated with beryllium and nickel. These tiles would be installed at the JET fusion device in Culham, U.K., in the frame of the ITER-like wall project.

Dr. Lungu is a member of the European Physical Society, the Romanian Physics Society, and the Romanian Trybology Association.

Andreea Roxana Lupu was born in Bucharest, Romania, on April 17, 1978. She received the B.Sc., M.Sc., and Ph.D. degrees in neurobiology from the University of Bucharest, Bucharest, in 2004, 2006, and 2009, respectively.

She started her vocational work experience between 1999 and 2000. When following studies with Fundeni Hospital Nursing School, she was a Nurse with D. Danielopolu Institute for Normal and Pathological Physiology, Bucharest. Later, between 2004 and 2006, she was a Biologist Research Assistant with the Cantacuzino Institute (Immunomodulation Laboratory) where she became a Scientific Researcher in 2006. Since 2008, she has been a Scientific Researcher with the National Institute for Laser, Plasma and Radiation Physics, Bucharest. Over the last five years, she worked and published in the fields of immunology and biophysics. Her general main interest topics concern effects and mechanisms for drug immunomodulation in inflammation and pain, design of new dosing technique in immunology, and study of cold-plasma–living-cell interactions.

Dr. Lupu is a member of the Romanian Immunology Society and the Romanian Society of Biochemistry and Molecular Biology.

Mariana Osiac was born in Schitu, Romania, on October 22, 1971. She received the B.Sc. and M.Sc. degrees in laser spectroscopy from the University of Bucharest, Bucharest, Romania, in 1995 and 1997, respectively, and the Ph.D. degree in plasma physics and spectroscopy from Ernst-Moritz-Arndt Greifswald University, Greifswald, Germany, in 2003.

During 1996–1999, she was with the National Institute for Laser, Plasma and Radiation Physics, Bucharest, as an Assistant Researcher. From 1999 to 2002, she was with the Institute for Low Temperature Plasma Physics, Greifswald. During 2003–2004, she held a Postdoctoral position at Ruhr University Bochum, Bochum, Germany. Since 2005, she has been an Associate Professor with the University of Craiova, Craiova, Romania. Her general main interest topics in the last five years are plasma spectroscopy, thin films, and pulsed laser deposition.

Dr. Osiac is a member of the Romanian Physics Society.