PECULIARITIES OF UV RADIATION FROM HOLLOW CATHODE DISCHARGE PLASMA USED FOR STERILIZATION OF MEDICAL ARTICLES

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Influence of air and water molecules detached from the chamber walls under ion impacts on UV radiation from hollow cathode discharge plasma in oxygen, nitrogen, and air is studied. Dependencies of UV radiation intensity and dose on the discharge glow duration, working gas type and pressure, and the discharge power are determined.

PACS: 52.80.Hc, 52.70.Kz, 52.25.Os

1. INTRODUCTION

A novel technique for sterilization of medical instruments by gas discharge plasma offers many promising features (efficiency, reliability, low temperature of processed items) in comparison with conventional sterilization methods – dry or moist heat, chemical treatments by EtO. It was shown in [1,2] for the first time that in the case of gaseous plasma generating media main role in the sterilization of open surfaces is performed by ultraviolet radiation of the plasma. In subsequent years, different aspects of both generation of UV radiation by the plasmas of low pressure discharges, and action of this radiation on the microorganisms were studied in multiple proceedings. However, all researches of the sterilization efficiency in different wavelength ranges of VUV/UV radiation from a discharge plasma were performed at stable and controlled system parameters, that is with permanent in time component content of a gas mixture, its pressure / gas flow, etc. In case of actual plasma sterilizers based on low pressure discharges the situation is somewhat more complicated. Ultimate parameters of a sterilizer are minimum sterilization time (more correctly, minimum time from loading to unloading of processed items), and the design simplicity. These requirements to the device are in a certain contradiction with experimental conditions listed above, because through working chamber evacuation with removal of atmospheric air residuals (N_2, O_2, water vapor) is required for providing stable and controllable content of required gas mixture. And the last can be don only at the expense of prolongation of evacuation time and the use of high vacuum pumps.

Experimental researches of sterilization efficiency by UV radiation under actual conditions, that is with varying in time component content of the gas mixture in the discharge due to atmospheric air admixtures to working gas (nitrogen, oxygen, argon) were performed in [3]. It has been determined that higher inactivation efficiency by UV radiation of the discharge plasma on oxygen is due to peculiarities of its emission spectra, particularly, high radiation intensity in wavelength range ≈ 215…230 nm.

Due to that, purpose of our work consisted in the study of peculiarities of generation of UV radiation by hollow cathode discharge plasma in nitrogen, oxygen and air with O_2, N_2 and H_2O admixtures.

2. DESCRIPTION OF SETUP AND METHODS

The experiments were performed at setup which was described in details in [2]. Plasma was formed by means of direct current discharge inside cylindrical chamber with 260 mm diameter and 420 mm length (which simultaneously served as hollow cathode of the discharge). Oxygen, nitrogen and ambient air were used as working gases. Specific power introduced into the discharge was varied in a range of 0.0025…0.0125 W/cm^3. Temperature of water cooled chamber walls was about 20°C.

Measurements of spectrum dependencies of the plasma UV radiation in wavelength range of 200…300 nm on the discharge glow time t_g were performed by means of spectrometer SL40-2-2048USB (SOLAR TII, Ltd). At the measurements, the end of quartz waveguide of the spectrometer was located in a plane which corresponded to placement of Petri dishes with *Bac.subtilis* spores during medical-biological researches.

Due to fact that experimental studies of decontamination efficiency of *Bac. subtilis* spores were performed with the use of UV radiation with essentially different spectrum shape, in this proceeding the method of determining effective irradiation fluence for studied sample was used which enabled correct comparison of the results obtained with the use of mentioned UV sources. Essence of the method consisted in "weighing" spectral irradiance values for each used type of UV radiation.

At determination of "weighing" function, first of all, results of works [7, 8] devoted to studies of DNA molecule absorption and efficiency of bactericidal action of UV radiation on the microorganisms in dependence on the radiation frequency were taken into account. It has been shown in [4] that DNA absorption spectrum in considered wavelength range (≈180…300 nm) represents superposition of broad absorption bands having maxim at about 190 and 260 nm due to electron excitation of diene and triene fragments of DNA molecule chain. In experiments with *Bacillus subtilis* spores [5] it has been shown that spores inactivation action spectra (i.e. dependence inactivation rate vs UV wavelength) has more complicated behavior in comparison with DNA absorption spectrum. In wavelength range 50…300 nm the spores inactivation rate has peaks at ≈ 150 nm, ≈ (220…230) nm and ≈ (260…270) nm. (Such difference is most likely due to absorption of ra-

ВОПРОСЫ АТОМНОЙ НАУКИ И ТЕХНИКИ. 2010. № 4.
Серия: Плазменная электроника и новые методы ускорения (7), с.234-239.

diation by the structures surrounding DNA – exosporium, plasma membranes, protoplast, etc.). In our work we used "weighing" function (Fig.1) obtained by multiplying inactivation action spectra for *Bac. subtilis* spores type RCF from [5] and transmission curve of KU-1 filter.

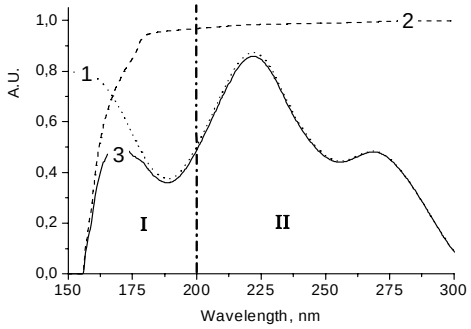

Fig.1. 1 – Bac. subtilis spores inactivation action spectrum; 2 – transmission curve of quartz KU-1 filter with 3-mm thickness; 3 – "weighing" curve, obtained by multiplying of the spores inactivation action spectrum and transmission curve of KU-1 filter

Dash-dot vertical line represents bottom boundary (200 nm) of spectra measurements of UV radiation from the discharge plasma. (It should be noted that, at the use of "weighing" function of the inactivation spectra for others spore types from [8], obtained values of "weighed" UV fluence rate E_w and fluence H_w of the radiation differ by no more than 15…20%).

Procedure of accomplishing the experiments on sterilization / radiation spectra measurement was, as follows:

- the chamber was filled by air up to atmospheric pressure after glow of the discharge with particular parameters (working gas type, pressure, specific power in the discharge W_d);
- Petri dishes with *Bac. Subtilis* spores were unloaded from / loaded to the chamber (with about 10 minutes duration of the procedure), or the chamber was held at atmospheric pressure for the same time in case of UV spectra measurements;
- the chamber was evacuated by means of forevacuum pump down to residual air pressure of about 1 Pa;
- after that the chamber was purged by working gas at working pressure for 5…10 minutes;
- in subsequent, the discharge was ignited in the chamber with predetermined specific power W_d and the sterilization was performed, or the plasma UV radiation spectra were measured in dependence on the discharge glow time t_g;
- the discharge was turned off, the chamber was filled by ambient air up to atmospheric pressure, unloading / loading of Petri dishes was performed, or the chamber was held at atmospheric pressure for about 10 minutes;
- the same procedure of sterilization / UV radiation spectra measurements was repeated for other gas type, working pressure in the chamber, specific power in the discharge W_d.

Due to fact that prior to evacuation the chamber was held for a long time at atmospheric pressure, air and water vapor were adsorbed at the walls. Chamber evacuation and purge by working gases did not remove adsorbed particles from the walls completely (first of all, oxygen and water molecules), and due to that they were admixed with working gas at the expense of "knocking down" by fast plasma ions. Thus, the discharge glow occurred in working gas with admixtures of detached air and water molecules, at that content of these admixtures decreased with time t_g.

3. MEASUREMENTS OF SPECTRA OF UV RADIATION FROM THE DISCHARGE PLASMA IN A RANGE OF 200…300 nm

Fig.2 shows "weighed" spectrum intensity distributions of UV radiation obtained at the discharge glow with different working media.

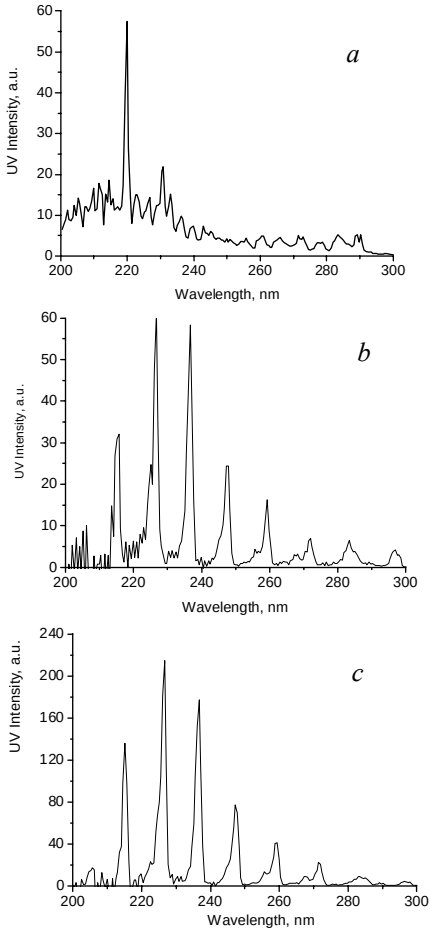

Fig.2. Distributions of "weighed" intensity of UV radiation over spectrum on different working media of the discharge: a – oxygen; b – nitrogen; c – air. Pressure P= 15.6 Pa, W_d = 0.08 W/cc, duration of discharge glowing t_d = 240 s

As one can see from analysis of these spectra, at oxygen use main contribution to UV radiation of the plasma occurs due to emission of the second negative system (SNS) O_2^+ ($A^2\Pi_u - X^2\Pi_g$) and Schumann-Runge system (SRS) O_2 ($B^3\Sigma_u^- - X^3\Sigma_g^-$), and at the use of ambient air and nitrogen due to emission of γ system NO ($A^2\sum^+ - X^2\Pi$). (Occurrence of radiation of γ system NO in the discharge plasma in nitrogen is due to presence of oxygen molecules at the expense of their detachment from the chamber walls under action of the fast plasma ions). One can see from comparison of Fig.2,a,b,c that in case of oxygen use main "weighed" power of UV

radiation is concentrated in spectrum range of ≈ 210…230 nm, whereas at the use of air and nitrogen power of UV radiation is spread over several bands in range of ≈ 210…260 nm.

As it was already noted above, at accomplishing the measurements component content of the gas in the chamber could somewhat vary at the time of the discharge glow t_g due to air and water vapor detachment from the chamber walls by flows of high-energy (W ≈ 400…600 eV) ions from the discharge plasma. Respectively, this effect can lead to dependence of UV radiation spectrum intensity and/or shape on time t_g. However, spectra measurements have shown that in a range of working pressure gas (oxygen, nitrogen and air) in the chamber of ≈ 4…25 Pa detachment of gases / vapors from the chamber walls has no significant effect on UV radiation spectrum shape in wavelength range of 200…300 nm. An exclusion is represented by the discharge in oxygen at ≈ 4…7 Pa pressure, when for initial 40…50 s of the discharge glow, in addition to radiation of O_2 and O_2^+ molecules, bands of γ system NO are also observed in UV spectrum. At the same time, value of "weighed" fluence rate E_w of UV radiation in this wavelength range essentially depends on both t_g , and working gas pressure in the chamber, at that these dependencies are different for various working gases (Fig.3,a,b,c).

Common behavior for all E_w dependencies consists in rapid growth of UV radiation intensity during initial ≈ 40…60 s, and after that their behavior in time depends on particular values of pressure and working gas type. This initial rapid growth of W_w is, first of all, due to pressure variations in the chamber at the discharge turning on. (At the discharge turning on, pressure in the chamber initially (for about 1 s) exhibits a jump by Δ P ≈ 2…3 Pa, and after that for ~ 40…60 s decreases monotonously down to predetermined pressure value in the chamber.) Plasma radiation intensity growth for this time period is, first of all, due to fact that pressure decrease in the chamber results in a growth of mean energy of the plasma electrons and, consequently, to increase of rates of elementary processes with participation of electrons.

In subsequent, (at t_g > ~ (40…60) s) when pressure in the chamber comes to its quasistationary value, behavior of E_w dependencies on t_g is, first of all, determined by ratios between concentrations of O_2, N_2 and H_2O molecules detached from the walls and working gas concentrations. Let us consider in more details W_w dependencies on time at t_g > (~ 30…60) s.

One can see from Fig.3,a that in case of the discharge in oxygen UV radiation fluence rate E_w at higher gas pressures is practically independent on t_g , and monotonously decreases with oxygen pressure increase in the chamber. However, at oxygen pressure of about 4 Pa behavior of E_w dependence on time is different – UV radiation intensity reaches its maximum at about 30 s of the discharge glow, after that it starts a decrease until its minimum at ≈ 150 s, and then it starts a growth again. (It should be noted that similar behavior of E_w dependence on time t_g is also observed at 7 Pa pressure, although its non-monotony is exhibited not so obviously). This effect may result from EEDF "depletion" in a range of ≈ (10…30 eV) due to losses of electron energy, first of all, for H_2O dissociation and OH excitation. And, since excitation cross sections of SRS O_2 and SNS O_2^+ are approximately in the same energy range, such EEDF "depletion" results in decrease of intensity of UV radiation from oxygen plasma.

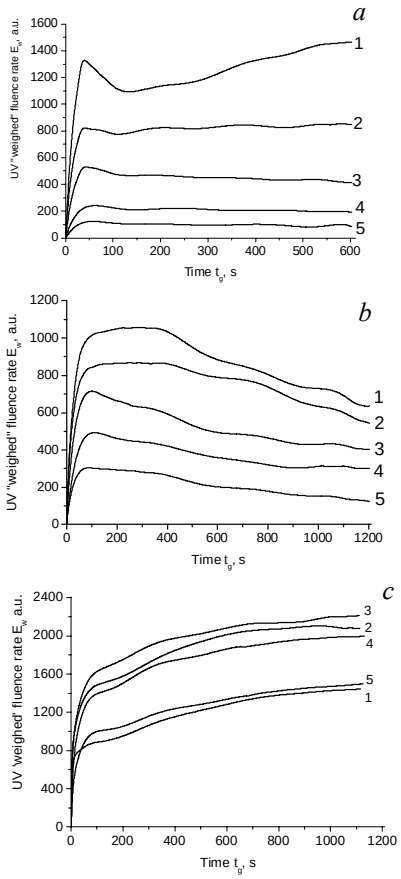

Fig.3. Dependence of "weighed" fluence rate E_w of UV radiation in wavelength range of 200…300 nm on the discharge glow time t_g at different pressure values P: a - oxygen; b - nitrogen; c - air. 1 - 4.5 Pa; 2 - 7.0 Pa; 3 - 11 Pa; 4 – 15 Pa; 5 – 21 Pa; W_d = 0.08 W/cc

Unlike the case of the discharge in oxygen, E_w value of UV radiation for the discharge in nitrogen continues a growth in time as well after ≈ 30…40 s of the discharge glow, however, with slower rate, and in subsequent, after reaching its maximum, it starts a decrease. At that, increase of nitrogen pressure in the chamber from ≈ 4 up to 20 Pa results in decrease of a time of reaching W_w maximum values from ≈ 300 s down to ≈ 100 s. Such behavior of E_w dependencies on t_g is, first of all, due to temporal variations of O_2 and H_2O concentrations in the chamber. For this reason, let us consider in more details main elementary processes which determine NO(A) concentration (and, consequently, UV radiation intensity of γ system NO) in our discharge plasma in nitrogen with O_2 and H_2O admixtures. It was determined in [6,7] that in a plasma of positive column of glow discharge in N_2-O_2 mixture main channel of NO(A) birth is represented by reaction:

$$N_2(A) + NO(X) = N_2(X) + NO(A), \qquad (1)$$
$$k = 6.6 \cdot 10^{-11}\ cm^3 \cdot s^{-1}.$$

In turn, NO(X) concentration in such discharge is mainly determined by ratio of rates of birth and death in reactions (2)-(4) and (5), respectively:

$$N2(X, v\geq 13) + O \rightarrow NO(X) + N(^4S), \quad (2)$$
$$k = 10^{-13}\ cm^3s^{-1};$$
$$N_2(A) + O = NO(X) + N(^2D), \quad (3)$$
$$k = 7\cdot10^{-12}\ cm^3s^{-1};$$
$$N(^4S) + O_2 = NO(X) + O, \quad (4)$$
$$k = 1.1\cdot10^{-14} * T * \exp(3150/T)\ cm^3s^{-1};$$
$$N(^4S) + NO = N_2(X, v\approx 3) + O, \quad (5)$$
$$k = 1.05\cdot10^{-12} * (T)^{0.5}\ cm^3s^{-1}.$$

Fig.4 exhibits experimentally measured dependencies of UV radiation intensity of γ system NO and weighed power of UV radiation W_w of our discharge plasma on oxygen content η in N_2-O_2 mixture. One can see that mentioned above dependencies show non-monotonous behavior – their initial growth changes to decrease at η ≈ 25%, at that radiation intensity of γ system NO in subsequent (at η ≈ (75…80)%) falls down practically to zero value, and E_w retains finite value due to contribution of oxygen UV radiation. Such behavior of γ system NO is determined by fact that the dependencies of $N_2(A)$ and NO(X) concentrations on oxygen content in the gas mixture also possess non-monotonous behavior, at that $N_2(A)$ concentration reaches its maximum value at lower η values [7].

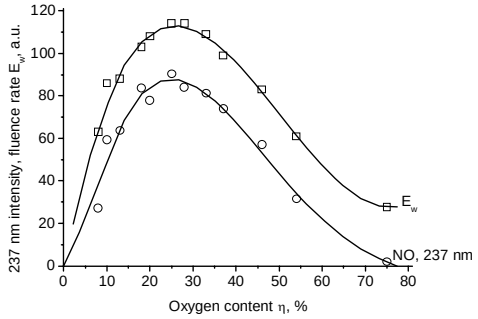

Fig.4. Dependencies of radiation intensity of γ system NO (λ = 237 nm) and "weighed" fluence rate E_w on oxygen content in N_2 - O_2 mixture. P = 8 Pa, t_g = 240 s, W_d = 0.08 W/cc

It also follows from Fig.4 that in case of our discharge in nitrogen E_w value (as it was noted above, it is actually radiation of γ system NO) after $t_g \geq 40…60$ s (that is, after establishing stationary value of gas mixture pressure in the chamber) should decrease in time, since concentration of molecular oxygen admixture in the discharge volume decreases in a process of the chamber evacuation. However, as one can see from Fig.3,b, such behavior of E_w is observed only at high nitrogen pressure in the discharge chamber, whereas at low pressure values E_w continues a growth until $t_g \approx 400$ s, and only after that starts a decrease. From our viewpoint, such behavior of E_w is due to water vapor influence (more exactly, OH(X) and H particles which arise at H_2O decomposition in the discharge plasma) on $N_2(A)$ concentration. Particularly, in [8-10] it was shown that $N_2(A)$ deactivation in reactions with OH(X) and H

$$N_2(A) + OH(X) = N_2(X) + OH(A), \quad (6)$$
$$N_2(A) + H = N_2H, \quad (7)$$

occurs with a very high rate constant – about $1\cdot10^{-10}\ cm^3\cdot s^{-1}$.

Thus, we see that water vapor adding to N_2-O_2 mixture results in decrease of concentration of $N_2(A)$ and, respectively, NO(A). In case of our discharge in nitrogen it means that E_w in each time point t_g is determined by "competitive" influence of O_2 and H_2O admixtures on NO(A) concentration. On one side, decrease of oxygen admixture in time should lead to diminishing concentrations of O and NO(X) (and in the last case NO(A), respectively). But, from another side, decrease of water vapor amount in the discharge promotes increase of NO(A) concentration due to lower expense of $N_2(A)$ to excitation of hydroxyl radicals OH(X). Thus, if for certain time rate of vapor removal from the chamber exceeds rate of oxygen leaving, it can result in increase of NO(A) concentration, even if quantity of NO(X) molecules in the chamber decreases at that. At low nitrogen pressure influence of mentioned above processes on NO(A) concentration and, consequently, on UV radiation intensity, is more evident due to higher relative contribution of water vapor admixture to total pressure in the chamber.

Let us consider now E_w behavior in case of the discharge in ambient air. As it was already mentioned above, UV radiation intensity W_w in this case increases monotonously in time at all used pressure values of ambient air. At the use of air as working gas, influence of oxygen admixture coming from the chamber walls on behavior of E_w dependence on t_g should be considerably less due to relative smallness of the amount of this admixture, as compared to oxygen amount in air. Due to that, NO(A) concentration (and, consequently, E_w value) in each time point should be mainly determined only by the processes of quenching $N_2(A)$ excited molecules by OH(X) and H particles. Thus, with a decrease of amount of water vapor amount detached from the chamber walls E_w value should grow up. Behavior of temporal dependencies of O_2 and H concentrations in our experiments is estimated by radiation of Schumann-Runge system O_2 and Balmer line of hydrogen H_α (radiation intensities of these particles are, in the first approximation, proportional to their densities, since their excitation occurs only by electron impacts). One can see from Fig.5 that the intensity of radiation of oxygen molecules diminishes in time rather quickly, and already at $t_g \approx 100$ s reaches its quasistationary value (that is, O_2 concentration in the chamber returns to the value corresponding the content in ambient air).

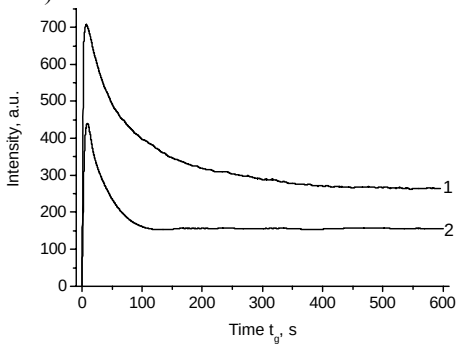

Fig.5. Dependencies of radiation intensities of certain lines of plasma particles of the discharge in ambient air on glow time t_g. 1 - H_α, 656.3 nm, 2 - O_2 (Schumann-Runge system), 219.4 nm. P = 8 Pa, W_d = 0.08 W/cc

At the same time, decrease of Balmer hydrogen line H_α radiation intensity (in other words, water vapor concentration) occurs considerably slower (practically "synchronously" with E_w value growth), thus approving the assumption expressed by us.

As it was already mentioned above, E_w depends not only on the discharge glow time t_g, but as well on gas pressure in the discharge chamber. One can see from Fig. 6 that in case of oxygen use E_w decreases monotonously with pressure increase in a whole used range of oxygen pressure, and curves of E_w dependencies for the discharges in air and nitrogen possess a maximum.

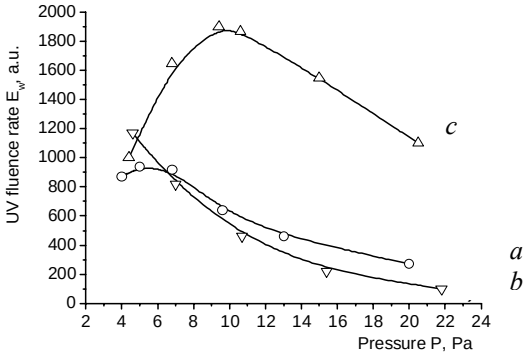

Fig.6. Dependence of "weighed" fluence rate E_w of UV radiation on pressure of gases: a - oxygen; b - nitrogen; c - ambient air at t_g = 240 s. W_d = 0.08 W/cc

It should be noted that 1) in spite of fact that main channel of NO(A) generation is reaction (1) with participation of only electrically neutral particles, concentration of the last is determined by processes with participation of electrons; 2) our previous researches have shown that, at constant specific power introduced into the discharge, plasma concentration is practically independent on pressure variation in a range of ≈ (4…20) Pa. Due to that, in all cases E_w behavior is determined by ratio of the rates of concentration growth for oxygen and nitrogen molecules and by decrease of the rates of reactions of their dissociation and excitation by electron impact at pressure increase (the last results in diminishing mean energy of the plasma electrons and, consequently, in decrease of rates of these reactions). That is, in case of the discharge in oxygen the reason of monotonous E_w decrease with pressure growth is due to fact that, in all range of P variation, excitation rates of SNS O_2^+ and SRS O_2 fall down faster than O_2 concentration grows up. Respectively, presence of maximum at E_w dependencies on P at the use of nitrogen and ambient air is due to lower, as compared to the case of oxygen, pace of diminishing the rates of oxygen dissociation reactions and excitation of nitrogen vibration levels. Let us consider in more details the reasons of such difference in paces of diminishing the rates of these elementary processes with pressure variation. In the first approximation, rates of the processes with participation of electrons $k \sim \sigma(\varepsilon)*f(\varepsilon)$, where $\sigma(\varepsilon)$ is the process cross section, $f(\varepsilon)$ is plasma electron energy distribution function. As it is known [11,12], threshold energies for cross sections of oxygen dissociation and excitation of nitrogen vibration levels are lower than threshold energies for cross sections of excitation of SNS O_2^+ and SRS O_2. And, since at decrease of mean energy of plasma electrons with gas pressure growth electron quantity in a range of cross sections of oxygen dissociation and excitation of nitrogen vibration level falls down slower than in a range of cross sections of excitation of SNS O_2^+ and SRS O_2, this results in lower rates of reactions of O_2 dissociation and excitation of N_2 vibration levels, as compared to the rates of reactions of excitation of O_2 and O_2^+. Here it should be also noted that in case of the discharge in nitrogen the behavior of E_w dependence on P is influenced by oxygen concentration decrease, as nitrogen pressure in the discharge chamber increases. Respective decrease of NO(X) concentration results in fact that E_w reaches its maximum values at lower gas pressure, as compared to the case of ambient air use.

It has been already determined that for all used gases fluence F_w of UV radiation practically linearly grows with t_g (see. Fig.7), at that F_w value for the discharge plasma in ambient air considerably exceeds UV fluence for the discharges in oxygen and nitrogen (which are close in their values) in the whole range of t_g variation.

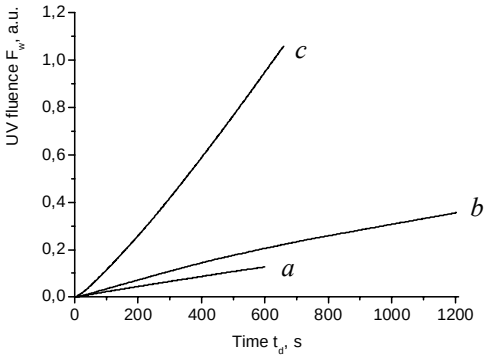

Fig.7. Dependencies of "weighed" fluence F_w of UV radiation on the discharge glow time t_g: a - oxygen; b - nitrogen; c - ambient air. W_d = 0.08 W/cc, P = 15 Pa

The experiments have also shown that at all gas pressure values UV fluence value grows up linearly with the increase of power introduced into the discharge W_d (Fig.8 represents respective dependencies for the case of the discharge in ambient air at pressure of 16.4 Pa).

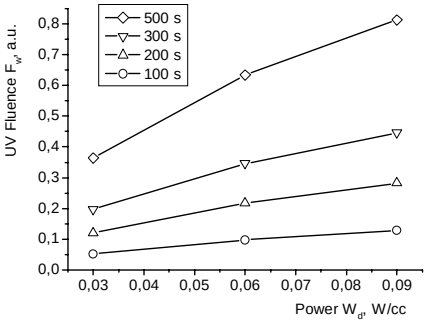

Fig.8. Dependencies of "weighed" fluence F_w of UV radiation on specific power in the discharge W_d at the use of ambient air for different time points t_g. P = 16.4 Pa

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Статья поступила в редакцию 31.05.2010 г.

ОСОБЕННОСТИ УЛЬТРАФИОЛЕТОВОГО ИЗЛУЧЕНИЯ ПЛАЗМЫ РАЗРЯДА С ПОЛЫМ КАТОДОМ, ИСПОЛЬЗУЕМОГО ДЛЯ СТЕРИЛИЗАЦИИ МЕДИЦИНСКИХ ИЗДЕЛИЙ

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Исследовано влияние примесей молекул воздуха и воды, оторванных от стенок камеры под действием ионных ударов, на УФ-излучение плазмы разряда с полым катодом на кислороде, азоте и воздухе. Установлены зависимости интенсивности и дозы УФ-излучения от длительности горения разряда, вида рабочего газа, его давления и мощности в разряде.

ОСОБЛИВОСТІ УЛЬТРАФІОЛЕТОВОГО ВИПРОМІНЮВАННЯ ПЛАЗМИ РОЗРЯДУ З ПОРОЖНИСТИМ КАТОДОМ, ЩО ВИКОРИСТОВУЄТЬСЯ ДЛЯ СТЕРИЛІЗАЦІЇ МЕДИЧНИХ ВИРОБІВ

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Досліджено вплив домішок молекул повітря та води, відірваних зі стінок камери під впливом іонних ударів, на УФ-випромінювання плазми розряду з порожнистим катодом на кисні, азоті та повітрі. Встановлено залежності інтенсивності та дози УФ-випромінювання від тривалості горіння розряду, виду робочого газу, його тиску та потужності в розряді.