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PHYSICAL ASPECTS OF THE CHEMICALLY ACTIVE MEDIUM FORMATION IN CHLORINE PLASMA UNDER PULSED DISCHARGE ENERGIZATION

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Abstract. *The research expands possible aspects of technological application of gas discharges in the technology of treatment and modification of various materials. Chemically active plasma allows intensifying conventional chemical processes at the expense of a more effective use of energy supplied to the system. By this approach all stages of the kinetic scheme of interacting particles technology in the plasma-chemical reactor become determinative. We study the processes observed at the signal switching fronts at pulse discharge power supply. Indeed, this mode of discharge combustion enables to achieve more effective utilization of reagents used in plasma chemical etching of different materials. Also we made the complex analysis of reasons causing appearance of transients at the front of signal switching in the discharge with periodical current variation. Moreover, we consider the relaxation of heavy charged and neutral particles in chlorine plasma.*

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Introduction

Nowadays low frequency discharges (LFD) are widely used in various practical and scientific applications. For example, a discharge at a frequency of 10 kHz is used in CO_2 lasers because this type of discharge, being in-between high-frequency discharges and DC discharges, has a number of fundamental features. There is a breakdown at the electrodes during the corresponding half-periods with the formation of a cathode layer but a weak low-frequency bias current does not cause current shorting at the electrodes which stabilises the laser performance [1, 2]. In addition, low-frequency power supplies for excitation of LFDs are significantly cheaper than pulsed or resonant microwave ultra-high frequencies-generators and are structurally simpler and more stable in operation.

On the other hand, the use of plasma for technological purposes is a very effective method of generating chemically active particles in plasma reactors. For example, halogen or oxygen

based plasma is an effective medium for the etching of various materials [3]. These gases are reactive themselves, but in addition, they can be enhanced in plasma by their dissociation to form atoms, which are known to have even better chemical properties [4]. It is also important to note that plasma chemical etching processes are often faster than in conventional chemical reactors because of the constant internal cleaning of the surface of the reacting materials by plasma ions. This mode is particularly effective in plasmas of electronegative gases [5, 6].

One new practical application of LFD is its use in defectoscopy to visualise mechanical and structural defects on the surfaces of high-grade hard materials [7].

It is important to note that electrons in low-frequency discharge are generated at the fronts of current pulses by periodic bursts, especially if plasma almost completely decays during the pause between current pulses and a voltage exceeding voltage of stationary discharge burning should be applied to ignite the discharge (breakdown of discharge gap). This experimental fact is described in [8]. In particular, it was shown that when studying the discharge ignition in long tubes (which were several times bigger than the electrodes themselves), the initial voltage pulse applied to the discharge system electrodes was higher than the voltage necessary to keep the discharge burning. At the same time there was another known effect called 'memory effect' [9]. Its essence is that after the first voltage pulse initiating plasma processes, the following pulses at discharge ignition fronts differ from the first one. In case of small pauses between the supply voltage pulses, the signal bursts at the leading edge of the voltage pulse are, as a rule, smaller than the first pulse. Otherwise they appear to be almost identical. An explanation of this fact is given in [10]. Thus, according to the authors of this paper, the main reason for this phenomenon is the increased ionization rate in the LFD. Indeed, at the initial moment of the voltage pulse the current in the circuit is small, and the voltage drop on the discharge gap is large. The field in discharge is much bigger than stationary one, which initiates processes in plasma with participation of charged particles (electrons, ions) that influence electric conductivity of discharge gap and chlorine dissociation at electron impact. Thus, "memory effect" is nothing but residual electric conductivity of discharge gap due to residual concentration of ionized plasma gas after a pause between current pulses.

This fact suggests that in this type of discharge the dissociation of a molecular plasma gas (e.g., chlorine) will proceed more efficiently than in a DC glow discharge. This fact may be useful in the case of chlorine plasma used for etching of materials. On the other hand, it is obvious that in LFD the excitation, ionization, and dissociation processes take place mainly in the combustion phase of the discharge, forming, together with recombination processes, stationary values of radicals, ions, and excited particles. In the pause phase of current pulses, the energy is spent exclusively on recombination processes. This fact makes it possible to use LFD for diagnostic purposes to study the recombination processes of chlorine atoms in plasma using the relaxation pulse method (RPM).

The main purpose of this work was to study the transients observed at the signal switching fronts during pulsed discharge feeding. This task is quite important, because characteristic times of active particle death in plasma can be tenths and hundredths of a second, i.e., they can be comparable in time with the period of current pulse repetition for low-frequency discharge.

Problem statement and description of the experimental plant

In this work we investigated LFD in chlorine, because, on the one hand, it is quite often used medium for plasma chemical etching of materials, and, on the other hand, in chlorine plasma it is possible to study independently the processes of heterogeneous recombination of atoms (without formation of products) and plasma chemical etching of different materials. In [11] we have already used RPM to study these processes. At the same time, sharp bursts of signal were recorded at the fronts of current pulse switching in several cases. It did not interfere with the main task – to obtain the spectral kinetic dependence of chlorine atom recombination in conditions of gas discharge combustion. It occurred only because the characteristic times of the investigated processes were longer than the times of the observed transients at switching the discharge on and off. In fact, these transients at the switching fronts were simply ignored, and the spectral kinetic dependences were extrapolated to the actual signal front. This approach is justified, however, if the processes under study have commensurate times with the signal ejections at the leading edge of the current pulse, then the implementation of the relaxation pulse methodology will prove to be difficult. This may become relevant when realizing RPM together with emission spectroscopy, which demands us to change the pause duration between the phases of discharge combustion up to comparatively small values or when studying recombination processes of radicals with short life times.

The experiments were conducted on the apparatus shown in Figure 1.

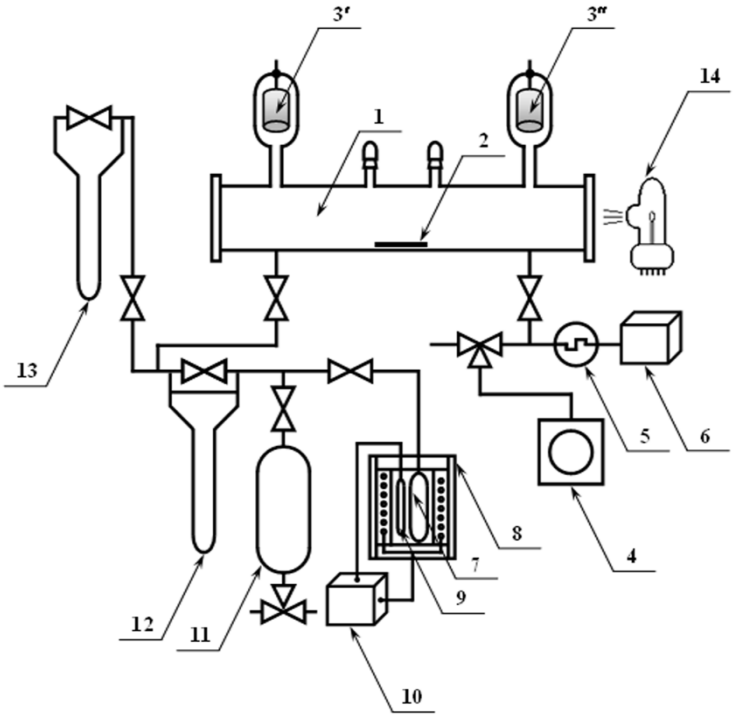

Fig. 1. Schematic of experimental setup: 1 - plasma-chemical reactor; 2 - sample under study (silicon); 3′ - electrode (cathode); 3″ - electrode (anode); 4 - rotary-oil pump VN-461; 5 - vacuum gauge VT-2; 6 - thermocouple sensor PMT-2; 7 - ampoule with $CuCl_2$ salt; 8 - furnace; 9 - chromel/alumel thermocouple; 10 - millivoltmeter Sh4501; 11 - chlorine storage tank; 12 - capillary oil rheometer; 13 - U-oil gauge; 14 - DDS-30 lamp

The main feature of the power unit we used for excitation of the discharge was that it allowed us to obtain a stable signal on the discharge tube with suppressed pulsations up to tens of microseconds. This made it possible to record transients in the discharge tube related to the

change of electrical conductivity of the plasma gap itself. It is important to note that the power supply design in the form of a DC source modulator was necessary, because one of the main assumptions for successful implementation of RPM, was the absence of integrating (differentiating) circuits in the circuit current, which are known to appear only on the AC component. As the main element of DC modulation circuit we have used generator pulse modulator lamp GPM-10. As a setting element of the circuit, we used a pulse generator of precise amplitude G5-75, which allows changing the length of pause t_p between pulses of discharge burning from 0.1 to 100 ms with discreteness not less than 1 ms (Fig. 2, *a*).

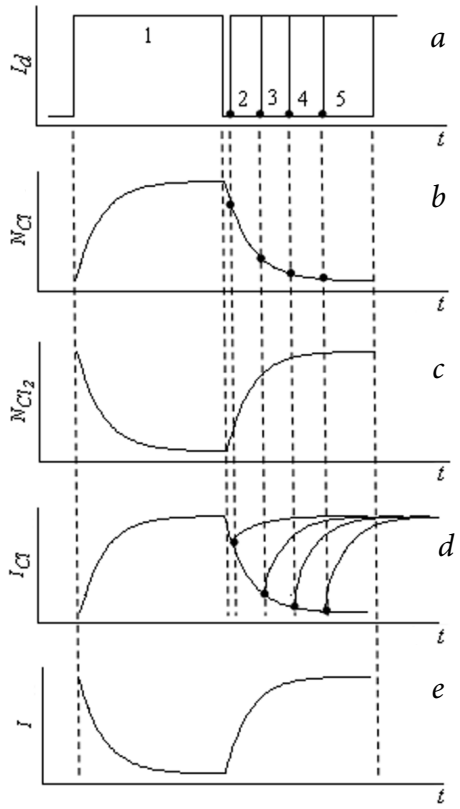

Fig. 2. Characteristic types of signals obtained in the course of the experiment: *a* - current pulses: time of discharge burning (1), pause times (2-5): t_{p1}-t_{p4}; *b, c* - dependences of concentrations of chlorine atoms and molecules at pulse discharge feeding; *d, e* - spectral kinetic dependence of atomic chlorine radiation and intensity of radiation passed through the discharge gap in LFD

For the above reasons, we have rejected the use of off-the-shelf switched-mode power supplies and power supplies based on resonant high-frequency circuits.

Results and discussion

RPM, together with emission and absorption spectroscopy were used to analyse the transients at the current pulse fronts in LFD. Spectral kinetic dependences of chlorine atom death in the discharge were obtained as a result of generalization of data on residual concentration of chlorine atoms in the discharge at different pause durations (Fig. 2, *b*). The relative change in chlorine atom concentration (Fig. 2, *d*) was determined using optical emission spectroscopy to study atomic chlorine at a wavelength of 452.6 nm (transition $5p^2p^0_{3/2} \rightarrow 4s^2p_{3/2}$). The possibility of using this transition in spectral quantitative measurements has been shown in [12].

Molecular chlorine concentration dynamics under pulse discharge feeding (Fig. 2, *c*) was controlled by molecular chlorine absorption by external radiation from DDS-30 lamp in wavelength range 330 nm (absorption spectroscopy) which corresponded to the maximum of molecular chlorine absorption band and linear section of lamp radiation. Fig. 2, *e* shows the intensity of the radiation passed from the lamp through the discharge gap when the discharge was pulsed. Photomultiplier detector PMD-39A together with the universal small-sized monochromator USM-1 was used as a sensor.

Fig. 3 and 4 show, respectively, the time dependences of the radiation intensity of atomic chlorine and the intensity of radiation passed through the glass plasma chemical reactor from the DDS-30 lamp.

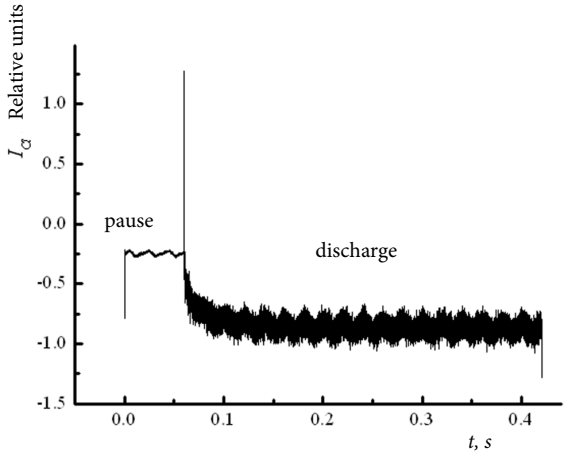

Fig. 3. Spectral kinetic dependence obtained using RIM together with emission spectroscopy, averaged over a set of single-type pulses, pause time 60 ms, period 420 ms

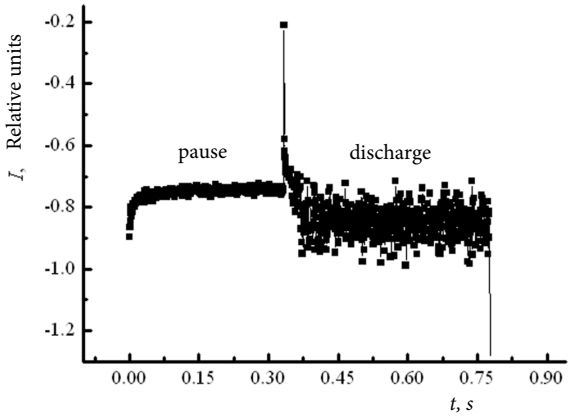

Fig. 4. Spectral kinetic dependence obtained using RIM together with absorption spectroscopy, averaged over a set of single-type pulses, pause time 333 ms, period 777 ms

In obtaining the data shown in Fig. 4, the absorption wavelength was varied relative to 330 nm so that the intrinsic emission of the discharge was kept to a minimum. All experiments were performed at a total system chlorine pressure of 100 Pa. The discharge current and plasma gas flow rate were maintained at 11 mA and 1.3 cm^3/s respectively. The pulse repetition period and the burn-up time of the discharge were chosen experimentally to minimise the effect of interference associated with the appearance of harmonics of multiples of 50 Hz (the frequency that feeds the mains circuit).

The dependencies shown in Figs. 3 and 4 were obtained as primary material necessary for the experimental determination of the recombination constant (probability) of atoms in different samples. As stated earlier, in earlier works transients at the switching fronts of the signals were ignored. In general, they did not interfere with the study of recombination kinetics in chlorine plasma, since in any case it was possible to fix the spectral kinetic dependence by extrapolating it to the beginning of the current pulse. In this work we decided to study experimentally fixed transients and possible reasons of their occurrence in more details. The point is that the times of transients and their type depended on the ratio of the discharge burning and pause times of LFD at an invariable pulse repetition period. Therefore, the circuitry cause of the transients should not be considered as the main cause. Thus, for example, the pulse repetition period of 420 ms (for RIM together with emission-spectrographic method), invariable for a

batch of experiments, was composed of the variable values of pause time (10-80 ms) and discharge burning time (410-340 ms), respectively. This suggested that the transients may be caused, on the one hand, by changing the concentrations of charged particles in chlorine plasma (electrons and ions) and, through the sequence of processes discussed in [13], by chlorine atoms being fixed at the leading edge of the voltage pulse. On the other hand, with a significant difference in the mobility of charged plasma particles (electrons and ions) the discharge gap should exhibit the properties of a reactive element. In this case, time oscillogram (Fig. 5), should have two phases: capacitive (1) and inductive (2), which are determined by the nature of motion of charged particles in plasma.

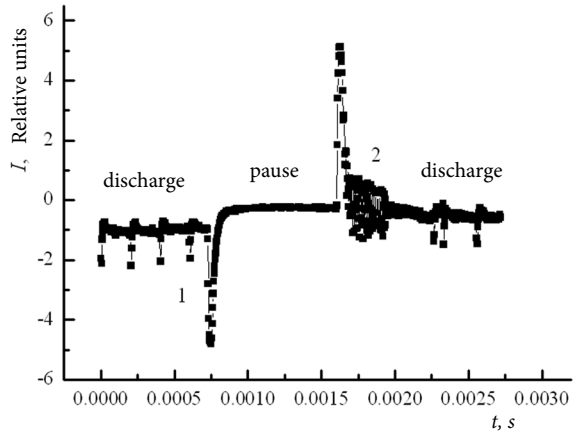

Fig. 5. Example of transients when the discharge is on - capacitive component (1) and when the discharge is off - inductive component (2)

It is not possible to state unequivocally in favour of a specific mechanism for the occurrence of transients. Most likely, the type of experimental dependencies can be explained by both of the above-mentioned factors. Thus, the data of Fig. 3 on radiation of atomic chlorine can be interpreted as electromagnetic induction arising on the reactive element of the circuit (discharge tube). The data on the absorption of external radiation by molecular chlorine (Figures 4-7) suggest a dual mechanism of transients.

Fig. 6 and 7 show detailed images of the transients in the LFD and Table 1 gives the characteristic area relationships for the pulses.

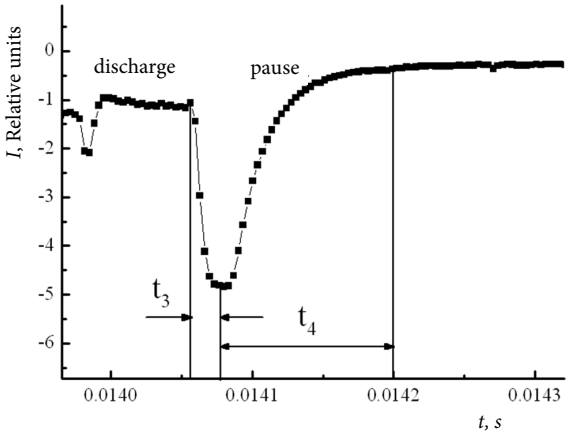

Fig. 6. Detailed depiction of the inductive phase of the transients in the LFD

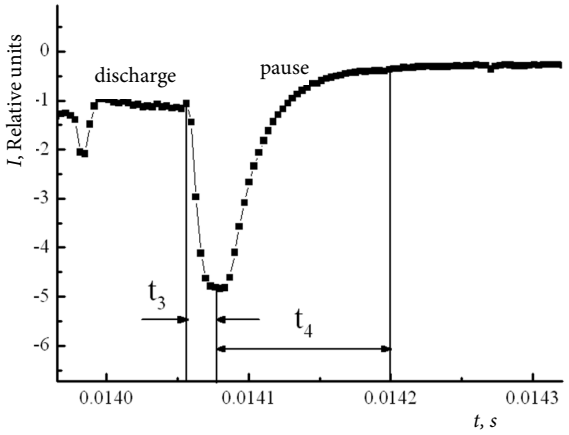

Fig. 7. Detailed representation of the capacitive transient phase in the LFD

Table 1. Values for inductive and capacitive pulse phase times in LFD. The designations of the time intervals t_1, t_2, t_4 correspond to Fig. 6 and 7; time interval t_3 remained unchanged at 0.019 ms

t_p, ms	t_1, ms	t_2, ms	t_4, ms
3	0.083	0.146	0.113
5	0.066	0.296	0.103
7	0.073	0.329	0.116
9	0.063	0.333	0.093
11	0,069	0,316	0.096
13	0,059	0,342	0.099
15	0.063	0.406	0.103
17	0.056	0.379	0.093
19	0.056	0.476	0.096
20	0.089	0.402	0.109
30	0.096	0.419	0.133
40	0.099	0.339	0.093
50	0.066	0.329	0.103
60	0.053	0.306	0.079
70	0.066	0.316	0.123
80	0.066	0.303	0.116
90	0.053	0.359	0.139
100	0.066	0.409	0.119

In this regard, we should expect the manifestation of resonant fluctuations of the transients themselves, similar to the wave nature of charge transfer in LFD [14]. Data of Figs. 8 and 9 confirm the development of such resonance phenomena in plasma. The discharge burning time between current pulses was chosen sufficient for yield of atomic chlorine concentrations to invariable values.

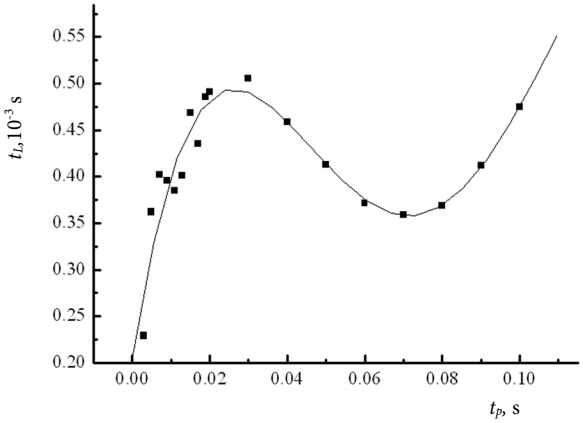

Fig. 8. Variation of transient development times for the inductive phase t_L of the LFD at different pauses of discharge combustion

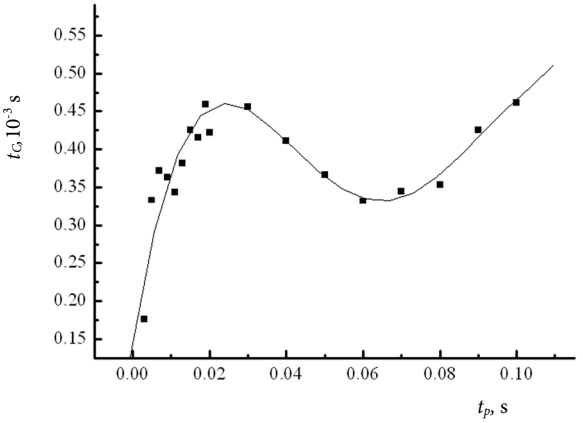

Fig. 9. Variation of transient development times for capacitive phase t_C LFD at different pauses of discharge combustion

Indeed, as the most probable cause of nonlinear transients at switching fronts of signals in LFD is the specificity of relaxation of gas discharge particle concentrations. Prediction of the character of relaxation processes, determination of characteristic times of their course requires analysis of kinetics and mechanisms of all processes influencing the balance of formation and death of particles of one or another variety. In this paper a generalized analysis of experimental results is presented to explain resonance processes at switching fronts in LFD, primarily based

on changes in the electron concentration in the decaying plasma during the pause between current pulses. In addition, relaxation of heavy charged and neutral particles is considered.

In a continuously burning discharge after some time after its ignition some stationary concentrations of neutral and charged particles, determined by the balance of their formation and death rates, are established. In pulsed discharge it is necessary to consider different influence on balance of particles in plasma both the active part of a pulse and a pause between them, and depending on pulse duration and repetition rate it is possible to establish quasi-equilibrium concentrations differing from each other.

In gaseous discharge all main processes are initiated mainly by electrons, therefore, the evolution of electrons in different phases of current pulse should be considered first. The build-up of electron concentration in the initial phase of the current pulse occurs because of ionization of neutral particles by electron strikes and can be described by an equation of the form:

$$n_e = n_{e_0} e^{k_i N t}, \tag{1}$$

where n_{e_0} is the initial electron concentration level.

k_i is the ionization rate coefficient;

N is the concentration of ionizable particles;

t is the current time.

The value of $1/k_iN$ is a characteristic ionization time and according to our estimations for the conditions of low-pressure discharge in chlorine is about 10^{-6}-10^{-7} s. Consequently, at pulse duration of the order of 160 ms the change in concentration of heavy particles in the main phase of current pulse occurs at unchanged electron concentration. However, initial stage of plasma formation at discharge switching on can be accompanied by pulse ejections of electron component.

When the discharge is switched off, one of the important points is the question of plasma deelectronization between pulses. Analysis of the relaxation kinetics of nonequilibrium gas-discharge plasma requires the joint solution of balance equations for all plasma particles. In this case, relaxation of electron energy is of prime importance, since it is under the action of electrons that most plasma processes are initiated. For the average energy of electrons E_e, we can write down:

$$\frac{dE_e}{dt} = \nu E_e(\delta_e + \delta_i), \tag{2}$$

where υ is the collision frequency of electrons with heavy plasma particles;

δ_e, δ_i is the average fraction of energies lost by electrons in elastic and inelastic collisions.

From expression (2) the following expression for the electron energy relaxation time follows:

$$\tau_{E_e} = 1/(\nu(\delta_e + \delta_i)). \tag{3}$$

In low-pressure chlorine plasma $\delta_i >> \delta_e$ and is 10^{-2} - 10^{-3} s. At the plasma gas pressure from 100 Pa and higher $\upsilon \geq 10^{10}$ s^{-1}, that corresponds to $\tau_{E_e} \leq 10^{-7} \div 10^{-8}$ s values. This is essentially less than electron concentration relaxation time [15]. Therefore, the process of changing of the electron concentration in time in decaying plasma can be approximately considered on

the assumption that the relaxation of the electron energy has already finished, and their mean energy is determined by the gas temperature.

In general, a decaying plasma is characterised by three main channels of electron death:

1. Diffusion to the walls followed by death on the surface.
2. An electron-ion recombination in volume.
3. Adhesion to gas molecules.

In the presence of negative ions in plasma, diffusion of charged particles is characterised by a free diffusion mechanism (in contrast to an ambipolar mechanism, typical for plasmas practically devoid of negative ions). The effective diffusion coefficient of electrons in plasma with negative ions can be found from the expression [13]:

$$D_{ef} = D_{+}\left(\frac{1+\gamma+2\beta\gamma}{1+(\mu_{+}/\mu_{e})(1+\beta)+\beta\mu_{-}/\mu_{e}}\right), \quad (4)$$

where D_+ is the diffusion coefficient of positive ions;

$\beta = n_-/n_e$ - relative concentration of negative ions;

$\gamma = E_e/E_g$ is the ratio of the average energies of the electrons and molecules of the gas;

μ_+, μ_-, μ_e - mobilities of positive, negative ions and electrons respectively.

Assuming from above mentioned considerations the value of γ = 1 and setting $\mu_+ = \mu_- = 7.5 \cdot 10^{-4}$ m^2/(V·s) and $\mu_e / \mu_+ = 250$, which is applicable with sufficient accuracy for estimates for many heavy gases including chlorine, the diffusion times of electrons in chlorine plasma containing negative ions were obtained. For the estimations we assumed that $D_+ = 2.3 \cdot 10^4$ cm^2/s. Results show that for our experimental conditions and a discharge tube radius of 1 cm τ_D varies from $2 \cdot 10^{-3}$ to $2 \cdot 10^{-5}$ s when β is varied from 0 to 100. However, similar calculations lead to the general conclusion that diffusion provides appreciable plasma de-electronisation times only at small discharge tube pressures and radii.

Among the electron-ion recombination processes of interest is dissociative recombination:

$$Cl_2^+ + e \rightarrow Cl + Cl,$$

which velocity coefficient at thermal energies of electrons is 10^{-5}-10^{-7} cm^3/s.

The lifetime of the electrons in this process is:

$$\tau_e = 1/(\alpha_{ei} n_{e0}), \quad (5)$$

where α_{ei} is the recombination rate coefficient;

n_{e0} is the initial concentration of electrons.

Simple estimates of the recombination lifetime τ_e by expression (5) assuming a n_{e0} range of 10^{10} to 10^{13} cm^{-3} and $\alpha_{ei} = 10^{-5}$ to 10^{-7} cm^3/s give values in the range 10^{-3}-10^{-8} s. These data allow us to conclude that dissociative electron-ion recombination is a fast process leading to plasma deelectronization in the pause between current pulses and cannot by itself explain the transient dynamics at the turn-off fronts of the pulsed discharge power.

Electrons sticking to chlorine molecules:

$$Cl_2 + e \rightarrow Cl_2^- \rightarrow Cl + Cl^-,$$

usually accompanied by dissociation of the latter, on the one hand, is an effective process of electron death in the decaying plasma, and on the other hand, it promotes the appearance of negative ions, which, in general, is characteristic of electronegative plasma-forming gases. This process contributes to the build-up of an excess concentration of ions in the plasma, which may lead to a reactive nature of the discharge gap impedance. The electron lifetime during the sticking process can be found from the expression:

$$\tau_{st} = 1/\sigma_{st} V_{\text{av}} N, \quad (6)$$

where σ_{st} is the adhesion cross-section, cm^2.

V_{av} is the average speed of the thermal motion of the electrons;

N is the concentration of chlorine molecules.

Both of these processes can cause a local concentration extremum of chlorine atoms.

Chlorine belongs to the type of electronegative gases for which dissociative adhesion requires no activation energy. The efficiency of the electron adhesion process to the molecules is characterised by the cross section value, which is estimated for chlorine as $\sigma_{st} = 1 \cdot 10^{-17}$ cm^2. Chlorine is not an effective gas in terms of dissociative adhesion efficiency, for example sulfur gas (SF_6) has electron adhesion cross-section equal to $1 \cdot 10^{-14}$ cm^2. Calculations by formula (6) for chlorine resulted in values of $\tau_{st} = 2 \cdot 10^{-7}$s.

Thus, the preliminary analysis shows that at small time intervals, which are characterized by chlorine plasma current pulse switching fronts, the processes of diffusion, recombination and sticking of electrons to plasma-forming gas molecules can affect the deelectronization of plasma decay in the current pause and through mechanisms of chlorine dissociation and ion formation with subsequent ion-ion recombination explain the causes of appearance of characteristic transient processes at current pulse switching fronts. To elucidate the role of a particular process in various conditions realized in the pulse discharge, specific calculations of time variations of electron concentrations are necessary, taking into account the main processes of their deaths. Such calculations can be carried out based on a generalized equation:

$$\frac{dn_e}{dt} = -\frac{D_e}{\lambda^2} n_e - \sigma_{st} V_{av} N n_e - \alpha_{ei} n_e n_+ \quad (7)$$

assuming that the energy of the electrons is close to the thermal energy.

Negative ions are effectively formed in chlorine and the condition of quasi-neutrality of the plasma must be written in the form $n_+ = n_- + n_e$. The electron recombination rate should be written as follows:

$$\frac{dn_e}{dt} = \alpha_{ei} n_e n_+ . \quad (8)$$

If $n_+ >> n_e$, which is characteristic of discharges of electronegative gases, then even complete recombination of electrons does not significantly affect the value of n_+. Therefore, the exponential form derived from expression (8) should be used:

$$n_e = n_{e0} e^{-\alpha_{ei} n_+ t} . \quad (9)$$

If we use expression (9), we obtain a sharper decrease of electron concentration and, consequently, a more effective production of the ionic component. Taking into account the fact,

that recombination processes of ions are much slower than the change of electron concentration, their contribution into explanation of the character of transients at the switching fronts can be interpreted in two variants. First, due to ion-ion recombination, which ensures quasi-neutrality of the plasma, excessive concentrations of chlorine atoms may be formed in the reactor in short intervals after the discharge is turned on (off). Secondly, the relatively high concentration of chlorine ions in the plasma, characterized by significantly lower mobility than electrons, creates reactivity conditions for the electric circuit in which the plasma reactor is included.

Conclusion and recommendations

Thus, the analysis of experimental data and estimates of characteristic times of charged particle death in LFD allow us to conclude that the main reason for appearance of transients at signal switching fronts is excessive concentration of electrons and ions generated in short intervals of discharge on/off time. The latter determines the unsteady reactive nature of the discharge gap, which can be characterised by inductive and capacitive components. The results obtained in this work, although estimated, may prove to be highly relevant and useful in the development of alternative approaches in the design of precision chemical reactors and the development of new technological processes.

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