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Probe methods of diagnostics of parameters of induction-coupled plasma maintained in a volatile halide gas mix

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In this paper, the results of the measurement of the local electron energy distribution function in an induction-coupled plasma discharge maintained in a flow of volatile halides at low-pressure are presented. A moveable electric probe with a short exposure time in plasma was used, which significantly reduces the erosive effect of the chemical active medium on the electrodes. Numerical methods of processing volt-ampere characteristics are discussed, which make it possible to identify the features of the electron energy distribution function in a high-energy region..

Keywords: plasma, electrical probes, halides, plasma chemistry.

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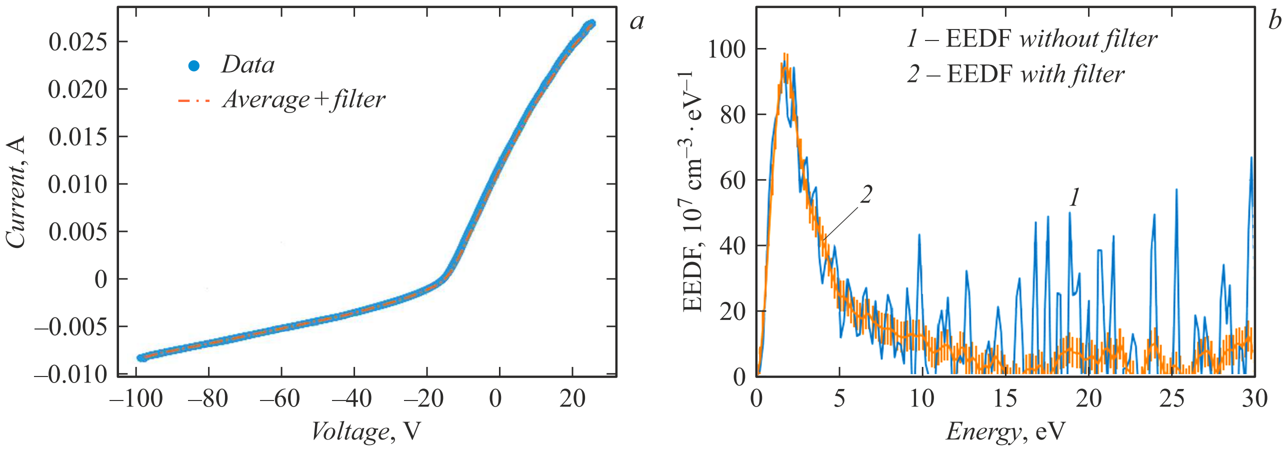
The development of diagnostic methods that allow the study of chemically active plasma parameters is an important applied task, the relevance of which is due to modern plasma chemical applications. For example, plasma decomposition of volatile fluorine and chlorine compounds is widely used in the synthesis of high-purity or isotope-enriched [1,2] substances. When modelling and optimizing plasma fusion processes, it is necessary to have information on the spatial distribution of the main parameters of the gas discharge [3]. The use of contact methods of plasma diagnostics makes it possible to measure local values of temperature and electron concentration [4], as well as to restore the shape of the electron energy distribution function (EEDF) [5]. However, the use of contact methods is limited due to the strong redox properties of chemically active plasma. For example, electrical probes injected into plasma can become contaminated with plasma fusion products that are produced during the decomposition of volatile halides. On the other hand, the use of non-contact methods [6] of diagnostics of plasma parameters of chemically active plasma also has a number of limitations. In particular, the deposition of reaction products on the surfaces of observation windows can distort the registration of emission spectra of the gas discharge. In addition, measurement by interferometric methods provides an averaged value of the electron concentration along the beam path [7]. To measure the spatial distribution of concentrations more accurately, contact methods of electrical probes [4] were used in this study.

The work presents a description of electrical probe [8,9] methods that have been used to diagnose chemically active plasmas. The main advantage of probe methods when using a periodic voltage is the speed of measuring the current-voltage curve (CVC) [4]. In the case of chemically active plasma, the probe interaction time with the aggressive medium should be minimized because the contact part is subject to contamination by plasma chemical reaction products, which leads to distortion of the probe curve. It has been experimentally checked that at repetition of measurements after 50 probe insertions into the region of chemically active plasma, which is approximately one minute of the total time of probe interaction with aggressive medium, the probe curve changes at the same parameters of discharge combustion and unchanged area of its measurement. This is assumed to be due to contamination of the probe as the contact surface becomes coated with plasma chemical decomposition products. After mechanical cleaning of the contacting part of the probe, the CVC becomes similar to the characteristic before the probe contamination. In addition, this method of a moving electric probe with a short exposure time in the plasma allows a probe curve to be obtained over the entire selected voltage range without measuring current readings at each particular voltage. The technique for an electric probe with a short exposure time in the plasma is as follows. With the help of a vacuum manipulator, the probe was brought to the discharge region. A signal, the period of which is less than the characteristic time of change of plasma parameters, and the value of the voltage amplitude is sufficient to manifest the effect of CVC saturation, was applied to the single probe. The probe curve was recorded at an applied signal frequency of 50 Hz and amplitude of 110 V. The probe was then withdrawn from the plasma region. The probe residence time in the plasma was determined by the speed of input and output using the manipulator and was 1 s. This method of operation allows levelling the effects associated with contamination of the contacting surface with reaction products. A filtering scheme for frequencies above 1 kHz was used in this work. Under similar conditions, a dual probe was used. The plasma parameters on the basis of the averaged CVC are calculated in a similar way to the calculations in [4,5]. The probe was made of a 0.3 mm thick molybdenum wire with



a — volt-ampere characteristic of a plasma consisting of a mixture of gases ($BF_3/H_2 = 1/3$, $(BF_3 + H_2)/Ar = 1/3$), at $p = 3$ Torr and discharge power 400 W.*Data* — current-voltage curve obtained from the initial data, *Average+filter* — moving average averaging and filtering.*b* — EEDF calculation without filter and with Savitsky – Golay filter after double differentiation (EEDF *with filter*). Discharge parameters: $BF_3/H_2 = 1/5$, $p = 3$ Torr, discharge power 400 W.

part of its length insulated by a ceramic sheath. The dual probe was two similar molybdenum wires with a spacing of 3 mm. Oscillograms were recorded using an AKIP 75444V oscilloscope with a bandwidth up to 200 MHz and a maximum sampling rate up to 1 GHz in two-channel mode. The first signal indicates the potential of the electrical probe relative to ground, and the second signal corresponds to the voltage drop across a resistor with a resistance of 510 Ω and is used to calculate the probe current. Probe diagnostics was carried out in the presence of an external electromagnetic field with a frequency of 13.56 MHz, which led to noise in the circuit elements, which were suppressed by digital filtering methods.

For initiation of the plasma discharge, the bench described in the [10] paper was used. The plasma discharge was ignited in a quartz tube with a diameter of 5 cm around which there was an inductor connected to an oscillator with a frequency of 13.56 MHz. The discharge was initiated in mixtures of Ar, H_2, CH_4 with volatile fluoride compounds (BF_3, GeF_4) in different proportions. Pressures were maintained in the range of 0.5 to 3 Torr. The discharge power ranged from 300 to 600 W. In order to ensure more reliable contact between the plasma and the antiprobe, which in the case of a single probe is a metal vacuum chamber, the inductor was displaced as close as possible to the metal chamber, but was isolated from ne. A discharge was ignited in the described above bench and then the concentration, electron temperature, and EEDF of the chemically active plasma at different plant parameters were calculated using electrical probe techniques. In the process of direct CVC measurements, the probe was located on the axis of the quartz tube in the centre of the inductor.

A single electric probe was used to calculate the shape of the EEDF. The EEDF shape was reconstructed from the experimental CVC of a single electric probe by its double differentiation [5]. The experimental CVCs were averaged over ten realizations using a moving average to minimize noise (see Figure, *a*). The resulting dependencies were numerically differentiated twice followed by smoothing using Savitzky's –Golay filter, and then the EEDF [5] form was calculated. The choice of an additional filter is due to noise oscillations arising during numerical differentiation (see Figure, *b*). Using a moving average or median filter resulted in distortion of the EEDF shape due to maximum shift. Filtering of the differentiated signal by the Savitsky – Golay method allows preserving the area, shape and height of „normal“ peaks. In addition, stepwise filtering at each iteration of the differentiation allows us to smooth out the noise in the numerical processing of the data, as well as to highlight the EEDF features in the high-energy region in the best possible way.

The electron concentration and temperature calculated from the reduced form of the EEDF [5] were $(4.3 \pm 1.7) \cdot 10^9\,\mathrm{cm}^{-3}$ and 4.5 ± 1.2 eV, respectively, at the following plasma discharge parameters: $BF_3/H_2 = 1/5$, $p = 3$ Torr, discharge power 400 W, and in the case of the „fast“ double probe method — $(10 \pm 3) \cdot 10^9\,\mathrm{cm}^{-3}$ and 5.5 ± 1.7 eV. In other modes of discharge combustion, the electron concentrations obtained by the „fast„ methods of single and double probes were of the same order of magnitude, and the electron temperature in the case of the double probe was slightly higher than in the case of the single probe. Based on the peculiarities of chemically active plasma, methods of moving electric probes with small exposure time can be used to process probe curves in chemically active plasma and to reconstruct the shape of EEDF.

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Conflict of interest

The authors declare that they have no conflict of interest.

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