Electron-beam plasma and its applications to polymer treatment in the forevacuum

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Abstract. We present our studies of the parameters of helium beam plasma. The beam plasma was generated by a powerful focused electron beam propagating through helium atmosphere at a pressure of 25 Pa. The beam power varied in the range 80 to 1000 W. We have determined the electron beam parameters that enable one to generate a beam plasma with a density greater than $10^{16}$ m$^{-3}$. Using the plasma ions, we have conducted treatment of polymer materials. It has been shown that the beam plasma treatment in the forevacuum range of pressure enhances the hydrophilic properties of fluoroplastic surfaces.

1. Introduction

Plasma is widely used today in microelectronics to manufacture semiconductor and quantum electronic devices [1]. Owing to its unique properties, the usage of plasma lies in the core of many technological processes, such as surface cleaning, anisotropic and selective etching, implantation and deposition of dielectric and semiconductor films [2]. The growing demand for microelectronic devices and their enhanced properties require accordingly improving the treatment technologies. Conventional methods of plasma generation are based on the use of electrical discharges operating in different frequency ranges. As of today, the most promising types of discharges used to generate plasma with high electron density are inductively coupled plasma (ICP) [3–5] and electron-cyclotron resonance discharge (ECR) [6–9]. Despite the high values of the plasma density and prospects of applications in plasma chemistry, such sources can inflict irradiative damages to the bombarded surface because of the presence of high-energy ions. Additionally, it is also important, when fabricating nanostructures, to minimize the electrical charging of the treated surface.

As an alternative method of creating a dense plasma, one may consider the electron-beam injection into a gas media [10]. By means of gas ionization, the electron beam generates the plasma whose properties can be controlled in a wide range of parameter values by varying the parameters of the electron beam, as well as by changing the type and pressure of the gas media. Such plasma is used in medicine to produce biocompatible coatings [11] and in microelectronics to etch thin atomic layers [12, 13]. As a rule, the beam generation and the plasma formation regions are separated by a pressure gap. The electron beam generation requires minimizing the flux of charged particles on the thermionic emitting cathode or, in the case of plasma cathode, diminish gas ionization in the source accelerating gap. On the other hand, generation of dense plasma requires using a pressure of tens of pascals. The pressure drop between the electron beam and the beam plasma generation regions may reach up to two orders of magnitude, thereby complicating the plasma generator design. Opposite to the conventional electron sources, the forevacuum plasma sources work in an isobaric regime, i.e. without a pressure
drop between the two generation regions [14]. Such sources are capable of processing both conductive and non-conductive materials [15, 16]. The absence of the pressure drop technologically simplifies the design and makes such sources attractive to generate the beam plasma and extract ion fluxes in order to use in the material treatment processes [17].

The purpose of the present work is to study the parameters of the beam plasma generated by a forevacuum plasma electron source and to use this plasma for processing polymer materials.

2. Experimental setup
The electron-beam installation used to generate beam plasma is shown in figure 1. It includes a vacuum chamber with evacuation system, a plasma electron source tailored to generate continuous electron beams in the forevacuum range of pressure. The electron source 1 generated an electron beam 2 with energy up to 20 keV and current up to 300 mA. Passing through the vacuum chamber, the electron beam created plasma 3. Upon exiting the vacuum chamber, the electron beam was captured by collector 4. The source was a three-electrode system that worked in a continuous regime. The hollow cathode and anode constituted the source discharge system. Electrons were accelerated by applying high voltage between the anode and the accelerating electrode. The source design also include focusing and deflecting magnetic coils. The source design and main parameters are discussed in more details in [18].

![Figure 1. Experimental setup. 1 – plasma source of a focused electron beam, 2 – electron beam, 3 – beam plasma, 4 – collector, 5 – double Langmuir probe, 6 – automated reading of the probe characteristics, 7 – fluoroplastic samples.](image)

The plasma parameters were measured using double Langmuir probe 5. Application of a bias voltage and reading of the probe characteristics were performed in an automated regime using registration device 6 with a built-in saw-tooth signal generator. The probe characteristic was processed in accord with the standard routine [19]. To prevent the probe from being hit by the electron beam, its design included a protective shield.

Fluoroplastic samples 7 were used as treatment materials. The samples had the shape of rectangular plates with dimensions 20 mm×20 mm×3 mm and were located in the beam plasma region at distances of 5, 10, 15 and 20 cm from the beam axis. The treatment by plasma ions lasted for 30 minutes. After treatment and removal from the vacuum chamber, the samples were analyzed with a scanning electron microscope and their hydrophilic properties were studied by the sessile drop method [20].

3. Experimental results and discussion
The electron temperature and plasma concentration were measured in helium atmosphere at 25 Pa for different values of the accelerating voltage and discharge current. Dependences of the electron
temperature on the discharge current for various values of the accelerating voltage are shown in figure 2.

\[ T_e (eV) \]
\[ I_b (mA) \]

**Figure 2.** Electron temperature vs. discharge current for various values of the accelerating voltage: 1 – 4 kV, 2 – 6 kV, 3 – 8 kV, 4 – 10 kV.

The experiments have demonstrated that increasing the beam current up to 100 mA and the accelerating voltage up to 10 kV leads to an insignificant change of the electron temperature in the beam plasma. Unlike electron temperature, the electron concentration depends more strongly on the beam current (figure 3). For the accelerating voltage of 4 kV, a five-fold increase of the beam current from 20 to 100 mA causes a five-fold increase of the plasma concentration from \( 0.47 \times 10^{15} \) to \( 2.3 \times 10^{15} \) m\(^{-3}\). On the other hand, for an accelerating voltage of 6 kV, the same five-fold increase of the beam current causes the concentration growth by an order of magnitude from \( 1.06 \times 10^{15} \) to \( 1.07 \times 10^{16} \) m\(^{-3}\) (figure 3, curve 2). Note that with increasing accelerating voltage, the magnitude, by which the concentration changes with increasing beam current, is different. For example, at the accelerating voltage of 8 kV, a five-fold increase of the beam current gives an increase of the concentration by 8 times. For the accelerating voltage of 10 kV, a similar increase of the beam current brings about a 6-time increase of the concentration. Such different influence of the accelerating voltage on the concentration increase is most likely related to the development of the beam instability. Upon reaching a certain energy, the beam electrons begin to build up plasma oscillations, thereby increasing energy of the plasma electrons and their ionizing capability.

\[ n_e \times 10^{15} \ (m^{-3}) \]
\[ I_b (mA) \]

**Figure 3.** Plasma concentration vs. discharge current for various values of accelerating voltage: 1 – 4 kV, 2 – 6 kV, 3 – 8 kV, 4 – 10 kV.
The pictures of the beam plasma glow taken for various accelerating voltages (figure 4) may speak in favor of such mechanism of the concentration growth. In the course of the experiments, we have detected a non-uniform glow of the beam plasma along the beam propagation path. Whereat the location of the brightest glow area depends on the accelerating voltage and the beam current. Increasing the accelerating voltage shifts the glowing area away from the electron source along the line of propagation (figure 4).

![Figure 4. Beam plasma glow at various values of accelerating voltage: a) $U_a = 4$ kV; b) $U_a = 8$ kV.](image1)

An increased value of the plasma concentration has enabled us to carry out treatment of polymer materials. As a polymer material, we used fluoroplastic plates, 3 mm thick with dimensions 20 mm×20 mm×3 mm. The fluoroplastic samples, located at distances from 7 to 19 cm from the electron beam with a step of 4 cm, were subjected to irradiation by the beam plasma ions. The accelerating voltage and the beam current were $U_a = 8$ kV and $I_b = 100$ mA, respectively. The treatment time was 30 minutes. The accelerating voltage was selected to ensure a high plasma concentration (figure 3) and at the same time a uniform distribution along the beam axis. Figure 5 shows pictures of the sample surfaces taken before and after treatment.

![Figure 5. Fluoroplastic surface before (left) and after (right) the treatment. The distance from the beam axis is 7 cm.](image2)
As seen from figure 5, the treated surface became smoother, which speaks for a possible surface polishing during the treatment process.

Figure 6 shows a bar chart of the wetting angle for various sample positions on the beam axis.

![Graph showing wetting angle vs. distance from the sample to the beam axis]

**Figure 6.** Wetting angle of the fluoroplastic surface vs. distance from the sample to the beam axis.

As seen from figure 6, as the substrate nears the beam axis, the fluoroplastic surface becomes more hydrophilic. At a distance of 7 cm from the beam axis, the wetting angle decreases almost by two times from 50 to 28 degrees.

### 4. Conclusion

It has been experimentally demonstrated that the concentration of the electron-beam plasma depends non-linearly on the beam current and the electron beam energy. For accelerating voltages less than 6 kV, the plasma concentration increases with increasing beam current. However, for the voltages higher than 6 kV, there is observed a more intense beam plasma production per the same increment of the beam current. The maximum concentration of the beam plasma $10^{16} \text{ m}^{-3}$ has been observed at the beam current 100 mA and the accelerating voltage 6 kV. Fluoroplastic treatment by such plasma ions increases hydrophilic properties of the material. After treatment, the wetting angles decreases by almost two times.

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