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CHARACTERIZATION OF EXTREMELY WEAKLY IONIZED HYDROGEN PLASMA WITH A DOUBLE LANGMUIR PROBE

KARAKTERIZACIJA ŠIBKO IONIZIRANE VODIKOVE PLAZME Z DVOJNO LANGMUIRJEVO SONDO

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Basic parameters of hydrogen plasma created in a large discharge chamber were determined using a double Langmuir probe. Plasma was created in a Pyrex cylinder with the diameter of 25 cm and the height of 80 cm by an antenna connected to a RF generator operating at the frequency of 27.12 MHz and the power of about 200 W. The antenna was a copper coil of 4 turns. The discharge chamber was pumped with an oil diffusion pump with the nominal pumping speed of 600 L/s backed by a two stage rotary pump with the pumping speed of 4.4×10^{-3} m^3 s^{-1}. The ultimate pressure of about 2×10^{-3} Pa was obtained in the vacuum system after pumping for few hours. A double Langmuir probe was galvanic separated from the mains and placed into the centre of the discharge chamber. The probe was made from 2 tungsten rods with a diameter of 1.2 mm and separated for 2 cm. The length of un-insulated part of the rods was 17.5 mm. Plasma parameters were measured at different pressures between 0.4 and 7.2 Pa. The electron temperature reached the maximum of about $kT_e = 3.5$ eV at the pressure of 1 Pa. The plasma density was slowly decreasing with increasing pressure and was of the order of 10^{15} m^{-3}, and the Debye length was rather constant at about 2×10^{-4} m. The results were explained by characteristics of an electrode less RF discharge in the E mode.

Keywords: plasma, hydrogen, double Langmuir probe, electron temperature, plasma density, Debye length

Z Langmuirjevo sondo smo določali parametre vodikove plazme, ustvarjene v velikem plazemskem reaktorju. Ta je bil narejen iz cilindrične cevi iz stekla pyrex premera 25 cm in višine 80 cm. Plazmo smo vzbujali z anteno, povezano z RF-generatorjem s frekvenco 27,12 MHz in močjo 200 W. Antena je bila narejena iz bakrene tuljave s 4 ovoji. Razelektritveno komoro smo črpali z difuzijsko črpalko z nazivno črpalno hitrostjo 600 L/s in povezano z dvostopenjsko rotacijsko predčrpalko s črpalno hitrostjo $4,4 \times 10^{-3}$ m^3 s^{-1}. Po nekajurnem črpanju smo dosegli končni tlak 2×10^{-3} Pa. Dvojna Langmuirjeva sonda je bila galvansko ločena od omrežja in vstavljena v sredino razelektritvene komore. Sonda je bila narejena iz dveh volframovih palic premera 1,2 mm, ki sta bili med seboj oddaljeni 2 cm. Dolžina neizoliranega dela palic je bila 17,5 mm. Parametre plazme smo merili pri tlakih med 0,4 Pa in 7,2 Pa. Temperatura elektronov je dosegla maksimum okoli $kT_e = 3,5$ eV pri tlaku 1 Pa. Gostota plazme je počasi padala z naraščajočim tlakom in je bila reda 10^{15} m^{-3}. Debyeva dolžina je bila dokaj konstantna okoli 2×10^{-4} m. Rezultate smo razložili z značilnostmi brezelektrodne RF-razelektritve v E-načinu.

Ključne besede: plazma, vodik, dvojna Langmuirjeva sonda, temperatura elektronov, gostota plazme, Debyeva dolžina

1 INTRODUCTION

Low pressure non-equilibrium plasma is nowadays widely used for treatment of solid materials on both laboratory and industrial scales. Interaction of plasma radicals with solid materials allows for modification of the surface free energy of different materials,[1–3] selective removal of particular compounds from the surface of composite materials,[4–7] modification of the surface roughness,[3,8–9] controlled destruction of organic materials[10–13] or surface functionalization,[14–24] and formation of non-equilibrium nanostructures on several materials.[25–38] Plasma is created in a suitable electrical discharge. Both magnetized and non-magnetized discharges are created using DC, AC, RF and MW power supplies. Depending on particular requirements, plasma is created in discharge chambers with different dimensions. Rather small chambers are usually applied for laboratory experiments. Namely, small systems are easy to control, almost free from electromagnetic interferences and cheap to build. A disadvantage of a small system is that plasma sustaining requires a certain density of charged particles. According to Paschen rules, the gas breakdown cannot occur in a small system at low pressure and low power. In some applications, however, very weak plasma is required. Since the Paschen rules prevent sustaining of such plasma in a small volume, one should either create plasma in a large system or use a flowing afterglow instead of plasma itself. Flowing afterglows are popular, but have an important disadvantage: since the reactive particles enter an afterglow chamber from a remote source, their density is very sensitive to properties of samples placed into the afterglow chamber. This effect is avoided by creation of plasma in a large chamber. In such a case, plasma can be created and easily sustained at pretty small discharge power and any particles that are lost at the surface of a sample are replaced by production in the chamber itself.

In order to understand interaction between plasma particles and solid materials, plasma should be characterized. A variety of methods have appeared for plasma characterization including titration,[39–41] optical emission and absorption spectroscopy,[42–46] mass spectrometry,[47–49]

catalytic probes[50–60] and electrical probes (often called Langmuir probes).[61–64] Basic plasma parameters are the density of charged particles, the electron temperature, the Debye length, plasma potential and floating potential. They can be all determined using electrical probes. A simple electrical probe is just a piece of metal immersed into plasma and connected to a DC power supply. The current against voltage characteristic is measured and the plasma parameters are calculated using an appropriate model. The probes perform well in stable discharges, but often fail in RF discharges due to stray effects. A piece of metal acts as a receiver and is self biased against surrounding plasma causing misleading results. An effective way to avoid such effects is application of a double electrical probe. In this configuration the probe is kept floating according to local space potential so the system is pretty unaffected by any stray effects as long as the entire probe circuit is galvanic separated from mains or any other metallic component. Such a probe was used at present experiments.

2 EXPERIMENTAL

A high vacuum experimental system was built to create very weak plasma. Schematic of the system is shown in **Figure 1**. The discharge chamber is a Pyrex cylinder with the diameter of 25 cm and the height of 80 cm. Plasma is created by an antenna connected to a RF generator operating at the frequency of 27.12 MHz and the power of about 200 W. The antenna is a copper coil of 4 turns. The discharge chamber is pumped with an oil diffusion pump with the nominal pumping speed on 0.6 m^3 s^{-1} backed by a two stage rotary pump with the pumping speed of 4.4 × 10^{-3} m^3 s^{-1}. Pressure is measured with a Pirani gauge that has been previously calibrated for hydrogen. The ultimate pressure of about 2 × 10^{-3} Pa was obtained in the vacuum system after pumping for few hours. After receiving a constant (ultimate) pressure the discharge vessel was filled with hydrogen to the desired pressure. Experiments were performed at different pressures between 0.4 and 7.6 Pa.

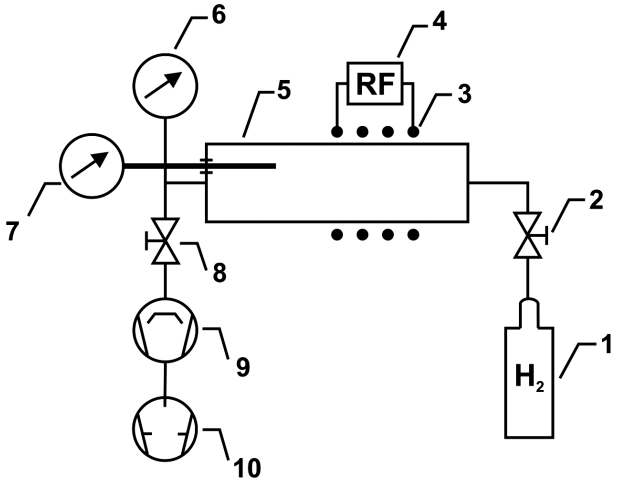

Figure 1: Schematic of the experimental setup. 1 – hydrogen flask, 2 – leak valve, 3 – copper coil, 4 – RF generator, 5 – discharge chamber, 6 – Pirani gauge, 7 – Langmuir probe, 8 – high vacuum valve, 9 – diffusion pump, 10 – mechanical pump

Slika 1: Shema eksperimentalnega sistema. 1 – jeklenka vodika, 2 – vpustni ventil, 3 – bakrena tuljava, 4 – RF-generator, 5 – razelektritvena posoda, 6 – Piranijev vakuummeter, 7 – Langmuirjeva sonda, 8 – visokovakuumski ventil, 9 – difuzijska črpalka, 10 – mehanska črpalka

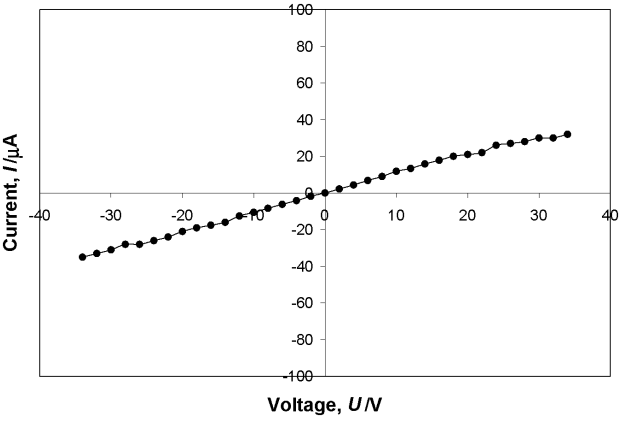

Figure 2: A typical characteristic of the small Langmuir probe
Slika 2: Značilna karakteristika majhne Langmuirjeve sonde

A double Langmuir probe was galvanic separated from the mains and placed into the centre of the discharge chamber. Two sets of probes were made: a small one with the length of 10 mm and the diameter of 30 μm and a much larger probe. The large probe was made from 2 tungsten rods with a diameter of 1.2 mm and separated for 2 cm. The length of un-insulated part of the rods was 17.5 mm. A typical characteristic of the small probe is plotted in **Figure 2**. The probe characteristic is rather linear instead of being similar to hyperbolical tangen as it should be according to literature. Obviously the small probe fails to operate properly and the reasons for this will be discussed later in the paper. Large probes were used for plasma characterization instead.

3 RESULTS

The probe characteristic was measured manually at different pressure. The voltage between the electrodes of the double probe was varied from –34 V to +34 V in steps of 2 V. The resulting current was measured by an ampermeter at each step. Some typical characteristics of the probe measured at different pressures are presented in **Figures 3 – 7**. Plasma parameters were then determined following the procedure suggested by Chen in his classical work.[65] The electron temperature was determined as[65]

$$kT_e = \frac{e_0 I_1 I_2}{\frac{dI}{dU}(I_1 + I_2)} \quad (1)$$

Here, k is the Boltzmann constant, T_e the electron temperature, e_0 the elementary charge, I_1 the saturated ion current on the first electrode extrapolated to zero net current, I_2 the saturated ion current on the second electrode extrapolated to zero net current, and dI/dU the first derivative of the curve at the inflection point. The values of I_1, I_2 and dI/dU are determined graphically as shown in **Figure 7** as for example. Once the electron temperature is known, the density of charged particles is calculated using the following equation:

$$N = \frac{I_1 + I_2}{e_0 A \sqrt{\frac{kT_e}{m_+}}} \quad (2)$$

Here, N is the density of charged particles in the vicinity of the probe, A the probe area, and m_+ is the positive ion mass (for H_2^+ ions it is 3.32×10^{-27} kg). The electron temperature and plasma density versus pressure calculated using Equations 1 and 2 are presented in **Figure 8**.

The Debye length is calculated as

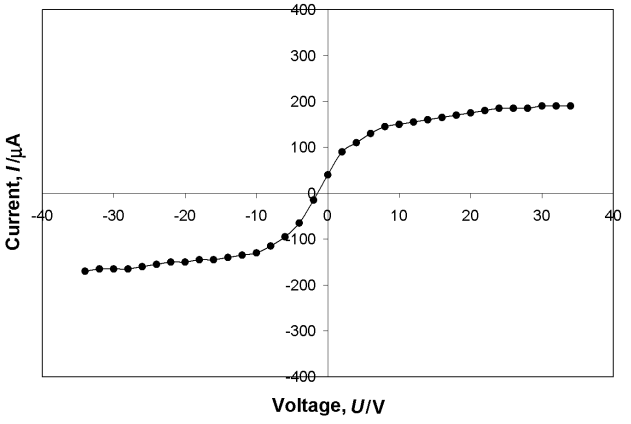

Figure 3: A characteristic of the large Langmuir probe measured at the pressure of 0.4 Pa

Slika 3: Karakteristika velike Langmuirjeve sonde, izmerjena pri tlaku 0,4 Pa

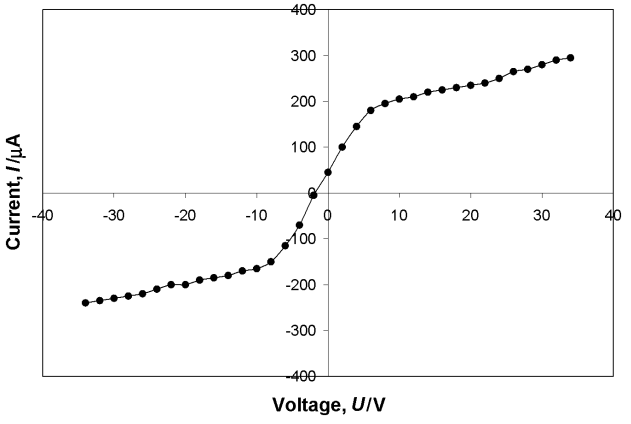

Figure 4: A characteristic of the large Langmuir probe measured at the pressure of 0.8 Pa

Slika 4: Karakteristika velike Langmuirjeve sonde, izmerjena pri tlaku 0,8 Pa

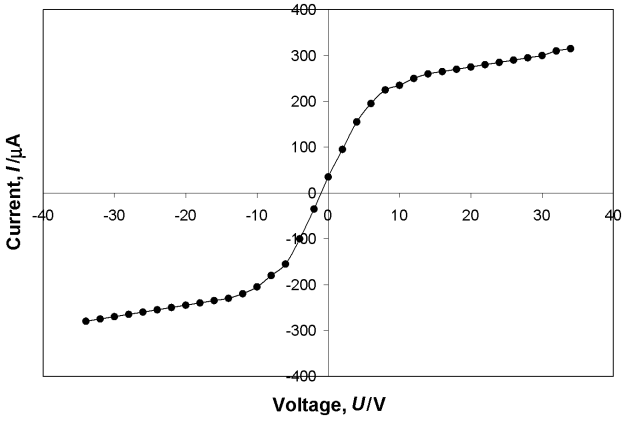

Figure 5: A characteristic of the large Langmuir probe measured at the pressure of 2.4 Pa

Slika 5: Karakteristika velike Langmuirjeve sonde, izmerjena pri tlaku 2,4 Pa

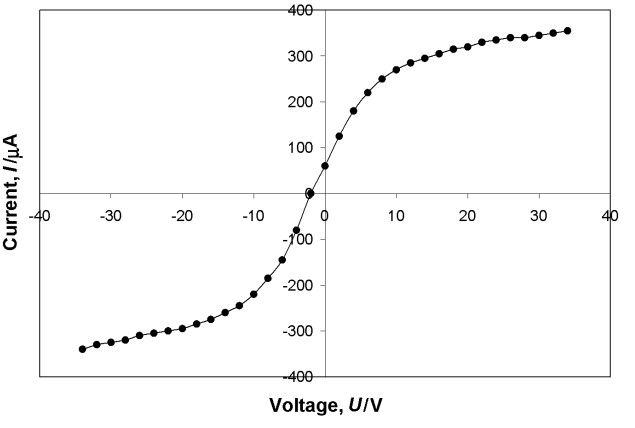

Figure 6: A characteristic of the large Langmuir probe measured at the pressure of 4.8 Pa

Slika 6: Karakteristika velike Langmuirjeve sonde, izmerjena pri tlaku 4,8 Pa

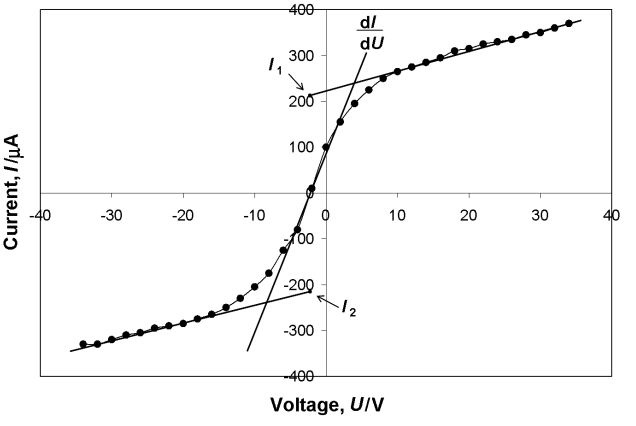

Figure 7: A characteristic of the large Langmuir probe measured at the pressure of 7.2 Pa

Slika 7: Karakteristika velike Langmuirjeve sonde, izmerjena pri tlaku 7,2 Pa

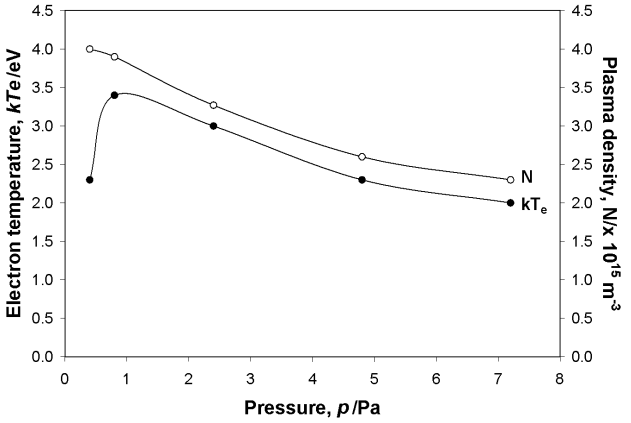

Figure 8: Variation of electron temperature and plasma density versus pressure

Slika 8: Spreminjanje elektronske temperature in gostote plazme v odvisnosti od tlaka

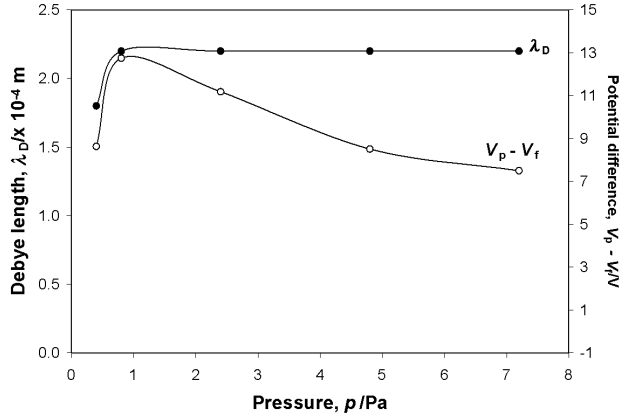

Figure 9: Variation of Debye length and variation of the difference between the plasma potential and the floating potential versus pressure

Slika 9: Spreminjanje Debyjeve dolžine ter spreminjanje razlike plazemskega in plavajočega potenciala v odvisnosti od tlaka

$$\lambda_{\mathrm{D}} = \sqrt{\frac{e_0 kT_{\mathrm{e}}}{N_{\mathrm{e}} e_0^2}} \tag{3}$$

Here, λ_{D} is the Debye length, and ε_0 the vacuum permittivity. Finally, the difference between the plasma and floating potentials is calculated from (4):

$$V_{\mathrm{p}} - V_{\mathrm{f}} = \frac{kT_{\mathrm{e}}}{2e_0} \ln\left(\frac{m_+}{2m_{\mathrm{e}}}\right) \tag{4}$$

Here, V_{p} is plasma potential, V_{f} floating potential, k Boltzmann constant, m_+ ion mass and m_{e} electron mass. The plots of the Debye length and difference between plasma and floating potentials versus pressure are presented in **Figure 9**.

4 DISCUSSION

Electrical probes usually fail in RF fields due to stray effects caused by electromagnetic interferences. Namely, any metal placed into the RF field acts as a receiver. This practical problem was minimized effectively by galvanic separation of the probe from any metallic part including the mains.

According to any literature, the probes should be made as small as possible in order to avoid drain of charged particles. While this criterion is particularly sever in the case of single probes that are biased also above the plasma potential to make proper reading of the electron temperature, it is less important in the case of a double probe. Namely, both electrodes of the double probe are kept well below the space potential so the drain of electrons is minimized. Still, an attempt was made to measure plasma parameters with a small probe with the electrode diameter of 30 μm. As clearly demonstrated in **Figure 2**, the small probe does not perform well in our case – the probe characteristic is rather linear instead of hyperbolical tangens. The strange behavior of the small probe is explained by taking into account the measured value of the Debye length (**Figure 10**). The Debye length is an order of magnitude larger than the small probe diameter. According to classical literature any object placed into plasma is surrounded by a sheath, i.e. an intermediate layer rich in positive charge between unperturbed plasma and an object. The sheath thickness is of the order of a Debye length and it increases with increasing bias of an electrode versus unperturbed plasma. The positive ions are well thermallized in unperturbed plasma and move randomly in unperturbed plasma. As they reach the sheath boundary they are accelerated towards the electrode and are finally collected by the electrode. The area from where the ions are collected is thus not the geometric area of the electrode but rather the sheath area. The sheath area increases rather linearly with increasing bias and so does the ion current. In the case when the electrode diameter is much smaller than the sheath thickness, the ion current therefore does not depend much on the electrode area but rather on the sheath area. Taking into account this consideration the observed characteristic of the small probe (**Figure 2**) is not surprising: the ion current is almost linear since the probe diameter is an order of magnitude smaller than the Debye length which is, as mentioned above, a measure of the sheath thickness. This is why the small probe fails in our plasma.

The situation is reversed in the case of the large probe (**Figures 3–7**). In this case, the Debye length is an order of magnitude smaller than the electrode diameter. The sheath thickness is thus much smaller than the electrode diameter so the characteristics are satisfactory. Still, a (rather linear) increase of the probe characteristics is observed in the ion saturation regime (at large biasing). This is due to the fact that the sheath thickness increases with increasing bias. Happily, the increase is not dramatic and can be taken into account following the suggestions of Chen.

Taking into account the upper considerations the value of the ion saturation currents used for determination of the plasma density (Equation 2) is somehow arbitrary since, strictly, the values should have been obtained at plasma potential. Happily enough, the slope of the characteristics in the saturated regime is much smaller than at the inflection point so the uncertainty in the plasma density is not big. Still, it is worth mentioning that the values of plasma density should be only taken as the first approximation.

5 CONCLUSION

The basic plasma parameters were measured in a rather large discharge chamber. The discharge power was kept pretty low at about 200 W. Pretty weak plasma was created at such experimental conditions. The plasma density was found to be of the order of 10^{15} m^{-3}. Such plasma is suitable for mild treatment of delicate materials that do not stand aggressive treatments. The plasma density slowly decreases with increasing pressure. Since the Debye length is much lower than the mean free path of hydrogen molecules at pressures applied, the sheath that forms around any object is collision less, so ions bombard the surface with the kinetic energy gained across the sheath. This energy depends on the voltage across the sheath and, according to **Figure 9**, on pressure, and is around 10 eV. The bombardment is thus really weak and should not cause any damage of samples immersed into the plasma due to kinetic effects.

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