

METHOD FOR DYNAMIC CONTROL OF NEUTRAL ATOM DENSITY IN A PLASMA CHAMBER

METODA ZA DINAMIČNO NADZOROVANJE GOSTOTE NEUTRALNIH ATOMOV V PLAZEMSKI KOMORI

Gregor Primc

Jožef Stefan Institute, Department F4, Jamova 39, 1000 Ljubljana, Slovenia
gregor.primc@ijs.si

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Systematic measurements of neutral oxygen atoms under various conditions and with the presence of a special active element (recombinator) were performed. Measurements of oxygen atoms will be used for future work on the regulation and dynamic control of neutral oxygen atoms. The experiment was performed in an experimental plasma reactor with a main and a side tube made from borosilicate glass. The neutral atom density was measured by a nickel-fiber-optic catalytic probe at a fixed position in the side tube. It has been found out that the density of neutral oxygen atoms depended both on the pressure of the oxygen gas and excitation power, but mostly on the presence and the position of the recombinator. The measured densities were of the order of 10^{21} m^{-3} , which is a characteristic value in glass discharge chambers. Such orders of density suffice for the processing of organic materials. The problem with organic materials is that interaction with oxygen atoms heats the material. Therefore, it is often necessary to decrease the atom density as soon as possible. As it is described in this paper we achieved the decrease in the atom density by using a movable recombinator. The recombinator has enabled us to select the appropriate atom density without having to change the discharge parameters.

Keywords: weak ionized plasma, oxygen plasma, fiber-optic catalytic probe, active control, neutral atom, atom source

Ob spreminjanju različnih parametrov in ob priostnosti aktivnega elementa (rekombinatorja) smo opravili sistematične meritve. Meritve bodo osnova za prihodnje delo na regulaciji in dinamičnem nadzoru nevtralnih kisikovih atomov. Za eksperiment smo uporabili eksperimentalni plazemski reaktor z glavno in stransko cevjo iz borosilikatnega stekla. Z nikljevo optično katalitično sondo na fiksnem položaju v stranski cevi, smo merili gostoto nevtralnih atomov. Ugotovili smo, da je gostota kisikovih atomov odvisna tako od tlaka, kot od vzbujevalne moči RF generatorja, predvsem pa od prisotnosti in položaja samega rekombinatorja. Gostote dosežene v eksperimentalnem reaktorju so bile reda velikosti 10^{21} m^{-3} , kar je značilna vrednost pri razelektritvi v stekleni cevi. Izmerjene gostote atomov so primerne za obdelavo organskih materialov. Obdelavo v plazmi spremljata dva procesa. Koristno prestrukturiranje površine vzorca, ki je bil izpostavljen plazmi, in nezaželeno segrevanje vzorca. Ravno zaradi segrevanja je pomembno poznavanje gostote kisikovih atomov, saj lahko previsoke koncentracije povzročijo uničenje vzorca. Kot je opisano v tem delu, nam je zmanjšanje gostote atomov uspelo doseči z uporabo rekombinatorja. Izkazalo se je, da lahko z rekombinatorji izberemo primerno gostoto atomov neodvisno od razelektritvenih parametrov, kar je še posebej pomembno pri obdelavi materialov v plazmi.

Ključne besede: šibko ionizirana plazma, kisikova plazma, optična katalitična sonda, dinamični nadzor, nevtralni atomi, vir atomov

1 INTRODUCTION

Nowadays weakly ionized highly dissociated gaseous plasma is widely used for treatment of different materials.¹⁻⁴ Namely, plasma treatment of materials features excellent quality, stability and above all ecological integrity. Different technologies often use oxygen, nitrogen or hydrogen plasma, especially as an alternative to environmentally unfriendly wet chemical treatment. Plasma is mainly used for surface cleaning,⁵⁻⁹ activation of organic materials,¹⁰⁻²² selective etching of polymers and polymer composites,²³⁻²⁸ cold ashing of biological samples, and in modern medicine applications for sterilization of sensitive materials²⁹⁻³¹ and lately also for nanomaterial synthesis.^{32,33}

As already mentioned, material treatment may not always be environmentally friendly. It often requires a sufficiently reactive medium to induce chemical changes on the surface of a material. Wet chemical treatment presents a great burden to the environment, as it uses

acids, lye, solvents or liquid hydrogenated hydrocarbons, which cause problems, such as health hazards during transports, increased consumption of energy and other resources tied to the production and the purification of such chemicals, storage and the industrial process itself and waste disposal after the process has been completed. Therefore, alternative methods, such as plasma treatment, are needed to minimize the impact of harmful chemicals on the environment.³⁴

When treating different materials it is very important to know the density of plasma particles in the vicinity of a treated sample.³⁵ Method and treatment intensity are highly dependent on particle flux density on the surface of the sample. Furthermore, diverse gradients of concentration may appear in the processing chamber, so position of the sample is very important. Frequently a treated sample represents a strong sink for plasma particles, so that particle flux density on the surface of the sample is dependent on dimensional and material properties of the sample.³⁶ Therefore, it is important to know the exact

neutral atom density and being able to actively regulate it during treatment, independently of discharge parameters.

2 EXPERIMENT

The neutral atom density was measured in an oxygen plasma powered by RF generator. We used five different RF generator output powers from 200 W to 600 W (in 100 W steps) and four different pressures (50 Pa, 70 Pa, 90 Pa and 120 Pa). A special movable recombinator was used to control the neutral atom density in plasma reactor. At a given pressure we varied the position of recombinator relatively to the tip of the probe. Seven different recombinator positions were used: -7.5 cm, -2.6 cm, -1.3 cm, 0 cm, 1.3 cm, 2.6 cm and 4 cm (distance measured from the probe tip). And finally, at a fixed pressure and recombinator position we varied the RF generator power. Due to the hysteresis between E- and H-modes, we could not increase the power at exact 100 W steps, but rather adapted them during measurements.³⁷⁻³⁹

The experimental system, which was constructed to serve as a source of neutral oxygen atoms in the ground state at room kinetic temperature, is shown schematically in **Figure 1**. The vacuum system was pumped using a two stage rotary pump with a nominal pumping speed of $80 \text{ m}^3 \text{ h}^{-1}$. The pump was connected to a 4-cm-wide glass tube via a high-vacuum-compatible angle valve. The vacuum elements were equipped with KF40 flanges. The conductivity near 100 Pa was much larger than the nominal pumping speed. Therefore, the effective pumping speed at the entrance to the glass tube was almost the same as the pumping speed of the pump. An absolute vacuum gauge was mounted perpendicular to the 4-cm-wide glass tube (**Figure 1a**). The glass tube was made from borosilicate glass (Schott 8250, Mainz, Germany), which has a low coefficient for the heterogeneous

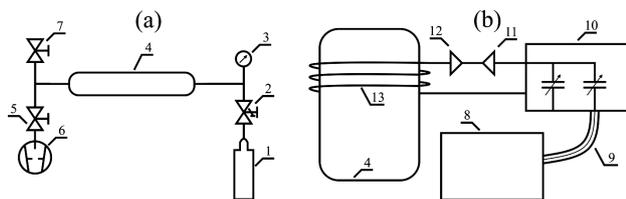


Figure 1: Part (a) represents a schematic of the experimental vacuum setup and part (b) represents a schematic of the electrical system. 1 – oxygen flask, 2 – precise needle valve used as an oxygen inlet, 3 – vacuum gauge, 4 – discharge tube, 5 – high vacuum valve, 6 – two stage rotary vacuum pump, 7 – air inlet valve, 8 – high frequency (13.56 MHz) RF power generator, 9 – coaxial cable, 10 – matching network, 11 – input power meter, 12 – output (reflected) power meter, 13 – RF coil.

Slika 1: Shema eksperimentalnega vakuumskega sistema (a) in shema električnega dela sistema (b). 1 – jeklenka s kisikom, 2 – precizni dozirni ventil za vpust plina, 3 – merilnik tlaka, 4 – razelektrivna cev, 5 – ventil, 6 – dvostopenjska rotacijska črpalka, 7 – ventil za vpust zraka, 8 – visoko frekvenčni (13.56 MHz) RF močnostni generator, 9 – koaksialni kabel, 10 – sklopitveni člen, 11 – usmerjevalni člen vstopne moči, 12 – usmerjevalni člen izhodne (odbite) moči, 13 – RF tuljava.

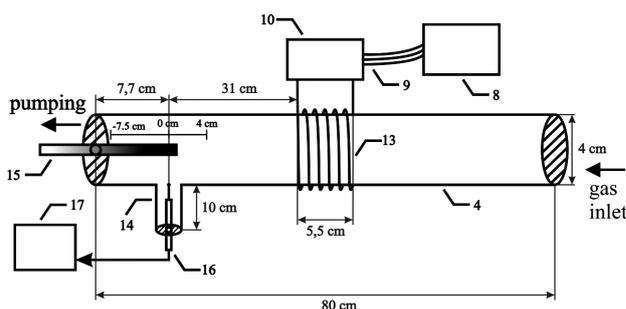


Figure 2: Technical drawing of plasma system. 4 – discharge tube, 8 – high frequency (13.56 MHz) RF power generator, 9 – coaxial cable, 10 – matching network, 14 – side tube, 15 – recombinator, 16 – nickel-fiber-optic catalytic probe, 17 – computer. The figure is not to scale.

Slika 2: Tehnična shema plazemskega sistema. 4 – razelektrivna cev, 8 – visoko frekvenčni (13.56 MHz) RF močnostni generator, 9 – koaksialni kabel, 10 – sklopitveni člen, 14 – stranska cev, 15 – rekombinator, 16 – nikljeva optična katalitična sonda, 17 – računalnik. Slika ni v merilu.

surface recombination of neutral oxygen atoms (less than 2×10^{-3}). The thickness of the glass tube was 2 mm; therefore, the inner diameter was 36 mm. The wide discharge tube also had a perpendicular side tube with the length of 10 cm and diameter of 1.5 cm (**Figure 2**). A manually movable copper rod with a high coefficient for heterogeneous surface recombination of oxygen atoms was used as a recombinator (**Figure 2**). The recombination coefficient of copper varies from 2.5×10^{-2} to 0.17.⁴⁰ The recombinator was placed into the main glass tube in the afterglow region. The position of recombinator was varied by moving it along the axis of the main discharge tube. On the other side of the discharge tube plasma was excited by an RF coil wound around the tube. The coil was water cooled, and the segment of the tube with the wounded coil was cooled by forced air. This cooling prevented a substantial amount of heating of the tube in the discharge region. The coil was connected to a RF power generator via a matching network and a coaxial cable (**Figure 1b**). The two power meters measured the input and reflected power into and from the coil. Usually some of the power applied to the system is reflected back to the generator causing coaxial cable and the generator itself to heat up. The matching network consisted of two high-voltage high-frequency variable vacuum capacitors that were used for adapting the impedance of "plasma-coil" system to the impedance of the remaining circuit, which is 50Ω .

A precise needle valve that served as an oxygen inlet was placed on the chamber and was connected to an oxygen flask via a standard copper tube, which had a diameter of 4 mm. Commercially-available oxygen that had a purity of 99.99% was used.

The density of neutral atoms was measured using a nickel-fiber-optic catalytic probe, which is described in detail in literature.⁴¹⁻⁴⁷ The probe was mounted 31 cm from the end of the coil in the side tube perpendicular to the main discharge tube. The absolute accuracy of the

probe is about 30%, but the relative sensitivity is extremely high, because it allows for the observation of even a small decrease in the oxygen atom density. The probe was coated by a nickel catalyst material, which (among some other materials) exhibits a constant recombination coefficient. It is important that the surface of the probe is properly prepared. To ensure stable surface recombination, the probe was exposed to a high flux of oxygen atoms, and consequently heated to a high temperature prior to systematic measurements. A thin oxide film was formed on the tip of the probe. Such thin film ensures for a repeatable and accurate measurements.

3 RESULTS

Measurements of oxygen atom density with nickel-fiber-optic catalytic probe were performed at various conditions (different pressure, power, and presence of recombinator). Even though the kinetic temperature of the gas is that of the surrounding gas (i.e. room temperature), the probe temperature quickly increased by several hundred Kelvin after the discharge was turned on. An example of such probe characteristic is shown in **Figure 3**. **Figure 3** shows the temperature of the fiber-optic catalytic probe as a function of time for five different excitation powers at a fixed pressure of 120 Pa and a fixed recombinator position at -2.6 cm. The maximum temperature was achieved after a few seconds to tens of seconds, depending on the discharge power. Such an increase in temperature is because of the very high probability of heterogeneous surface recombination for the oxygen atoms which cause heating of the probe. This probability was determined to be 0.27.⁴⁸

The plot of the maximum probe temperature as a function of the recombinator position is shown in **Figure 4** for the case of the minimum pressure (50 Pa) used and in **Figure 5** for the case of maximum pressure (120 Pa) used at our experiments. The parameter is real power. Real power is the power that ignites the discharge

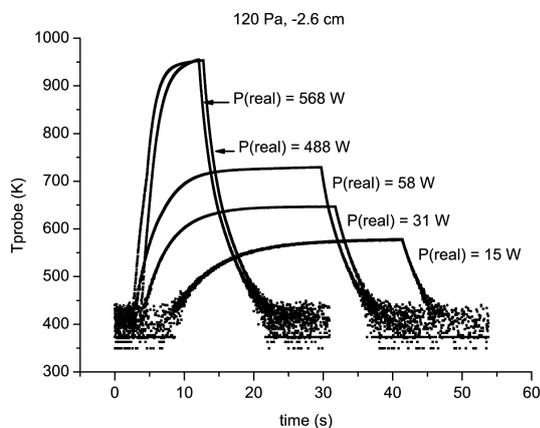


Figure 3: Temperature of the fiber-optic catalytic probe as a function of time at the pressure of 120 Pa and a fixed recombinator position
Slika 3: Temperatura optične katalitične sonde v odvisnosti od časa pri tlaku 120 Pa in fiksiranem položaju rekombinatorja

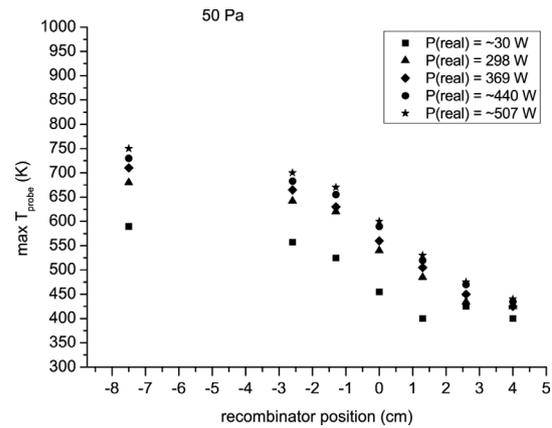


Figure 4: Maximum temperature of the probe as a function of recombinator position and the discharge power as a changing parameter. The pressure is 50 Pa.

Slika 4: Maksimalna temperatura sonde v odvisnosti od položaja rekombinatorja in razelektritvene moči. Tlak je bil 50 Pa.

and can be explained as a difference between the nominal or forward power and the reflected power ($P_{\text{real}} = P_{\text{forward}} - P_{\text{reflected}}$).

In **Figures 4 to 9** the recombinator position at -7.5 cm corresponds to a case when the recombinator is far away from the glow region i.e. far away from the source of oxygen atoms. While position at 4 cm corresponds to a case when the recombinator is placed very deep into the main glass tube and is therefore close to the glow region. We can see that the maximum probe temperature decreases when the recombinator is moved deeply into the glass tube. This decrease of maximum temperature is because of a lower flux of neutral oxygen atoms to the surface of the probe. The recombinator acts as a strong sink of oxygen atoms and, consequently, decreases the concentration of oxygen atoms in the discharge tube. Measuring of maximum probe temperature gives clear indication that we can control the density of oxygen atoms in plasma by changing the recom-

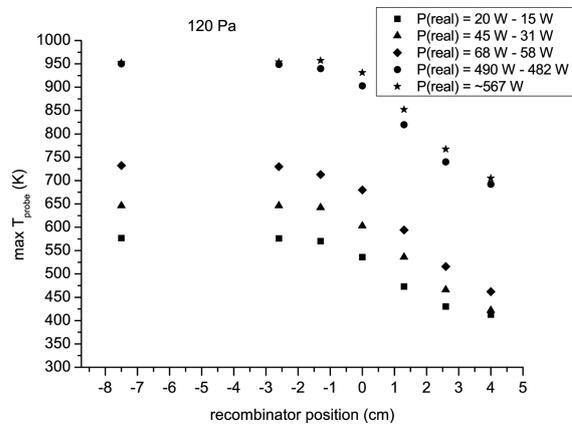


Figure 5: Maximum temperature of the probe as a function of recombinator position and the discharge power as a changing parameter. The pressure is 120 Pa.

Slika 5: Maksimalna temperatura sonde v odvisnosti od položaja rekombinatorja in razelektritvene moči. Tlak je bil 120 Pa.

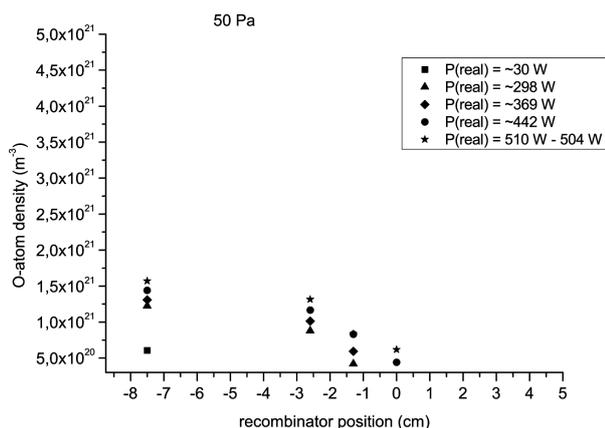


Figure 6: Density of neutral oxygen atoms at the probe position as a function of recombinator position and the discharge power as a changing parameter. The pressure is 50 Pa.

Slika 6: Gostota nevtralnih kisikovih atomov na področju sonde v odvisnosti od položaja rekombinatorja in razelektivne moči. Tlak je bil 50 Pa.

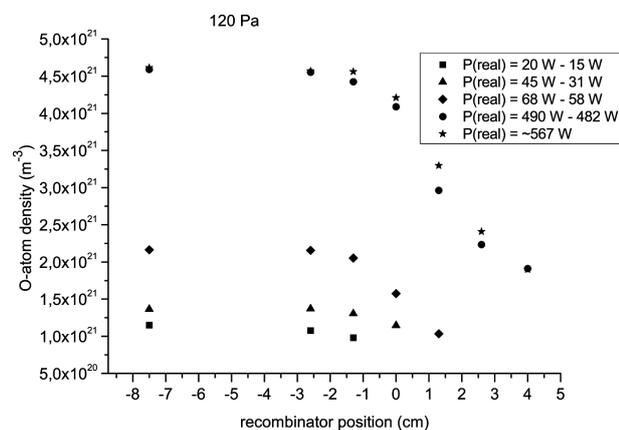


Figure 7: Density of neutral oxygen atoms at the probe position as a function of recombinator position and the discharge power as a changing parameter. The pressure is 120 Pa.

Slika 7: Gostota nevtralnih kisikovih atomov na področju sonde v odvisnosti od položaja rekombinatorja in razelektivne moči. Tlak je bil 120 Pa.

binator position. In order to get absolute values for the density of oxygen atoms in the vicinity of the probe we have measured the temperature decay just after turning off the discharge. The neutral oxygen atom density was calculated from the time derivatives of the catalytic probe according to the following equation:⁴⁸

$$n_0 = \frac{8mc_p}{\nu W_D \gamma A} \left(\frac{dT}{dt} \right) \quad (1)$$

where m is the mass of the probe tip; c_p is its specific heat capacity; ν is the average thermal velocity of oxygen atoms; W_D is the dissociation energy of an oxygen molecule; γ is the recombination coefficient of the oxygen atoms on the oxidized Ni surface; A is the area of the catalyst and dT/dt is the time derivative of the probe temperature just after turning off the discharge.

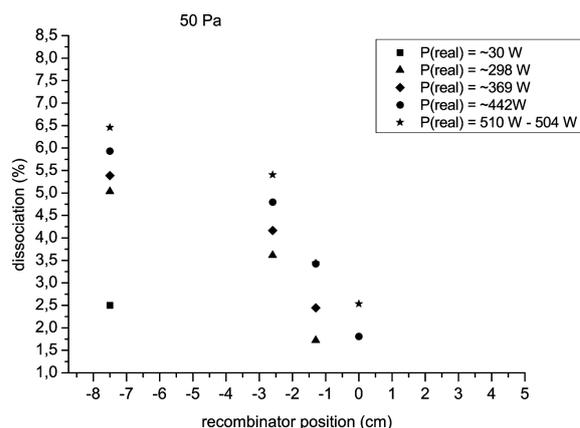


Figure 8: Dissociation fraction of the oxygen molecules at the probe position. The pressure is 50 Pa.

Slika 8: Disociacija kisikovih molekul na področju sonde. Tlak je bil 50 Pa.

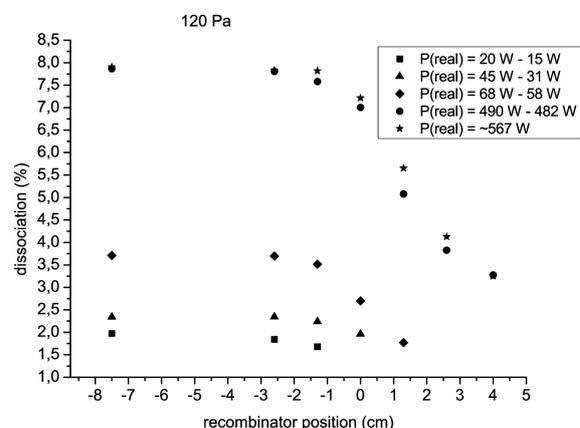


Figure 9: Dissociation fraction of the oxygen molecules at the probe position. The pressure is 120 Pa.

Slika 9: Disociacija kisikovih molekul na področju sonde. Tlak je bil 120 Pa.

Figures 6 and 7 show the oxygen atom density at the probe position using different discharge powers. Again, only results for the minimum pressure of 50 Pa (**Figure 6**) and the maximum pressure of 120 Pa (**Figure 7**) are shown. Corresponding dissociation fractions of the oxygen molecules are shown in **Figures 8 and 9**. The dissociation fraction was calculated from the known pressure before turning on the discharge and the oxygen atom density using the following equation:

$$\eta = \frac{n_0 k_B T_g}{2p_{O_2}} \quad (2)$$

where n_0 is the oxygen atom density; k_B is the Boltzmann constant; T_g is the gas temperature; and p_{O_2} is the oxygen pressure.

Note that some values are missing at graphs displaying the density of neutral oxygen atoms and dissociation fraction. At certain pressures, these values could not be measured because the probe signal was in the noise range.

4 DISCUSSION

Results displayed on **Figures 4 and 5** show a high temperature which at 600 W (in the case of 120 Pa) exceeds 900 K. At first glance such temperatures seem rather excessive because gas inside the chamber is at room temperature. Elevated probe temperature is interpreted by intensive recombination of neutral oxygen atoms on the surface of the probe. Oxygen plasma is rich in oxygen atoms. As we have already mentioned, the recombination coefficient of a glass discharge tube is low, therefore atoms recombine poorly on its surface. The excess of recombination is present on the surface of the probe and causes additional heating because of the nickel-catalyst coating on the tip of the probe. Because of this phenomenon the temperature of the probe is well above the temperature of the surrounding gas, which is approximately at room temperature.

The equilibrium temperature of the probe depends on the discharge power and oxygen pressure. In addition, the temperature of the probe largely depends on the recombinator position. Recombinator serves as a surface where extensive recombination of oxygen atoms takes place. As long as the position of the recombinator is far from the probe in the direction of the gas flow, it practically does not have any effect on the temperature of the probe. The directed gas velocity resulting in continuous pumping of the discharge tube is high enough to compensate the atom loss because of recombination reactions present on the surface of the copper recombinator. Moving recombinator closer to the probe noticeably influences on the oxygen atom recombination. In these circumstances the diffusion of the gas molecules and atoms is comparable to the directed velocity of the gas. The phenomenon is more distinctive when recombinator is pushed further inside the discharge tube passing the position of the probe. In this case, the recombinator causes a large decrease in the density of oxygen atoms in the vicinity of the probe. Therefore, the measured oxygen atom density is strongly dependent on the position of the recombinator. **Figures 4 and 5** show that the temperature of the probe decreases for approximately 50 K when we move the recombinator only for a centimetre. The recombinator therefore perfectly does its function, namely to decrease the neutral atom density independently of the discharge parameters. That was also the goal of the experiment; to quantitatively demonstrate that we may change the neutral atom density in any part of the discharge tube by moving the recombinator. When the recombinator is mounted deep inside the discharge tube (far right of the position of the probe) the temperature of the probe decreases rapidly and is, consequently, undetectable because of the noise picked up by the probe.

The temperature of the probe offers qualitative data about the neutral oxygen atom density. However, via measuring the time derivative of the temperature of the probe, quantitative data can be determined. The time

derivatives allow us to calculate oxygen atom density in the vicinity of the probe. The calculated densities are of the order of 10^{21} m^{-3} , which is a characteristic value in glass discharge chambers. Such high order of neutral atom density suffices for treatment of organic materials. The problem with organic materials is that the interaction with oxygen atoms heats the material. Therefore, it is often necessary to decrease the atom density as soon as possible. As it is described in this paper, we achieved the decrease in the atom density by using a movable recombinator. The recombinator has enabled us to select the appropriate atom density without having to change the discharge parameters.

As it is illustrated on **Figures 8 and 9**, the dissociation fraction of oxygen molecules is few percent. Such dissociation degree ensures the right processing of materials.

5 CONCLUSIONS

Systematic measurements of neutral oxygen atom density in cold weakly ionized oxygen plasma were performed. A movable copper recombinator was used in order to influence the density of neutral atoms. The densities at various pressures, high frequency RF generator powers and recombinator positions were measured by a nickel-fiber-optic catalytic probe at a fixed position. The measurements were carefully processed and displayed in several graphs, indicating maximum probe temperature, neutral oxygen atom densities and oxygen dissociation fractions, all as a function of recombinator position. Even though manually moving a recombinator seems somewhat an old-fashioned method, it proved to be extremely useful at manipulating neutral oxygen atom density independently of the discharge parameters. This is particularly important in material processing, especially when we are dealing with very sensitive organic materials. In order to avoid substantial heating and degradation of material it is significant to know the density of neutral oxygen atoms in the vicinity of the treated sample as excessive concentrations may harm or even destroy it. As shown in this contribution it is very easy to control the density of neutral oxygen atoms by moving recombinator deeply towards (or away from) the discharge.

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