Physical study of a microwave plasma source for agricultural applications

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Abstract: Low-pressure cold plasma may be of technical interest for large scale treatment in many fields. In this work, a microwave plasma discharge used for species decontamination was characterised by optical emission spectroscopy, microwave interferometry and Langmuir probe. The microwave power quantity transferred to the substrate through thermocouple measurements was determinated.

Keywords: Microwave plasma, Boltzmann plot, temperature measurements.

1. General

Low pressure and low temperature plasma is a non-thermal technology. The absence of toxics components and the large volume treatment capacity provide a good alternative to the conventional spices decontamination methods [1].

The process optimisation requires an excellent understanding and control of plasma characteristics. This includes the fundamentals plasma parameters (n_e, T_e, FDEE…), the type of species created (photons, reactive species…), the gas temperature, the substrate influence on the plasma for example.

In this work, the plasma chemistry has been investigated by optical emission spectroscopy. Probes measurements have been performed to, first, study the plasma parameters in several sustained conditions, and then, to understand the heating process of the substrate by the ionized gas.

2. Material and methods

The experimental set-up used for this study is presented in Fig. 1. It consists of a cylindrical stainless-steel vacuum chamber (35 L). The plasma is generated by a coaxial microwave source (Hi-Wave designed by SAIREM [2]). This source is water-cooled and powered by a 2.45 GHz solid state generator working from 1 to 200 W. The discharge chamber is pumped down by a vacuum system. Three mass flow controllers attached to the gas lines (Air, Ar/O_2, Ar) are used to keep the pressure treatment constant.

Plasma discharges have been characterized by Optical Emission Spectroscopy (Avaspec 2038-2 Avantes). Besides bringing out the plasma excited species, OES data were used to estimate the electron temperature T_e in argon plasmas through the Boltzmann plot method. Then, this result has been compared to those obtained by Langmuir probe measurements (Impedans Ltd.). Electron temperatures T_e were also determined in Air and Ar/O_2 discharges. Microwave interferometry was performed to map the plasma densities.

The temperature variation of the glass substrate depending on the microwave power injected was measured using a K-type thermocouple and calculated from the charged particle contributions reaching the substrate.

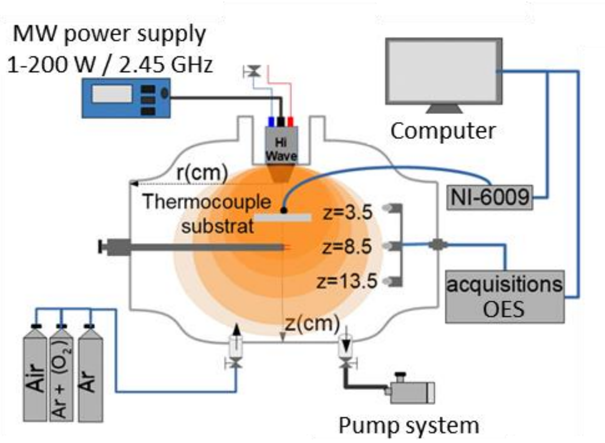

Fig. 1. Schematic view of the experimental set-up.

3. Results

3.1. Determination of T_e by the Boltzmann Plot method and Langmuir probe in argon plasma

Based on optical emission data, the Boltzmann plot is used to determine the electron temperature by inverting the slope of the following logarithmic functions:

$$\ln\left(\frac{\lambda_{ij} I_{ij} \sum_{i>j} A_{ij}}{hc\alpha_i A_{ij}}\right) = \frac{-E_i}{k_B T_e} + D \qquad (1)$$

where λ_{ij} and I_{ij} are the wavelength and the relative intensity of the emission line between the upper level i and the lower level j. $\sum_{i>j} A_{ij}$ represents the sum of the radiative transitions starting from the energy level considered. h is the Planck constant, c the light velocity, α_i a parameter representing the local thermodynamic equilibrium deviation, E_i the excitation energy of the level i and k_B the Boltzmann constant [3].

An example of the Boltzmann plots corresponding to an argon plasma sustained at 15 Pa and 150 W is depicted on Fig. 2. The wavelengths emissions taking into account are 751.46 ; 750.38 ; 731.60 ; 731.17 ; 714.70 ; 706.87 ; 687.95 and 516.22 nm. All the constants are available in the reference [4].

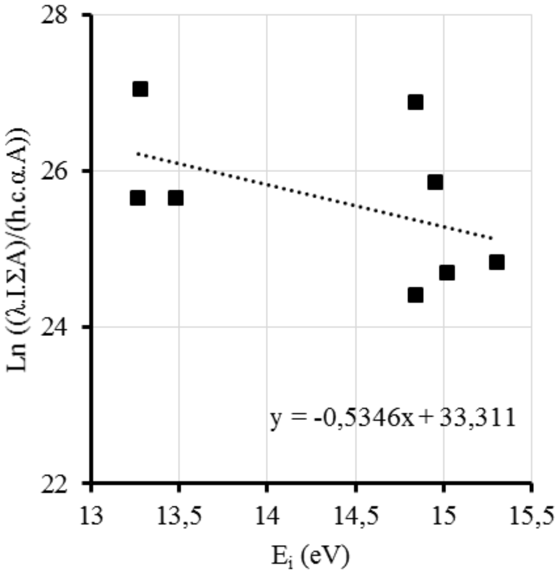

Fig. 2. Boltzmann plot in the case of argon plasma (15 Pa, 150 W).

Under this conditions, the slope calculation give a first approximation of T_e equal to 1.88 ±0.47 eV. The comparison with Langmuir probe data in Fig. 3 shows a good agreement with a variation of T_e between 2.0 and 1.3 eV depending on the gas pressure.

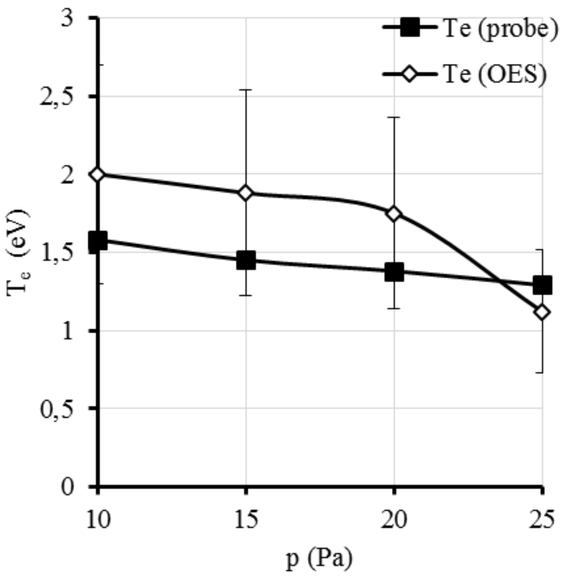

Fig. 3. Comparison between Boltzmann plot (T_e (OES)) and Langmuir probe (T_e (probe)) measurements in argon plasma generated at 150 W, at different gas pressures.

3.2. Determination of n_e by Langmuir probe and microwave interferometry

A motorized Langmuir probe was introduced at the axial position Z = 8.5 cm as shown in Fig. 1. This position corresponds to the distance between the microwave source and the sample carrier. Electron temperature and density values were collected along the radial axis *r* in plasma discharges filled by Air, Ar or Ar/O_2 (10%) mixture. Fig. 4 gives an example obtained at 150 W and 15 Pa.

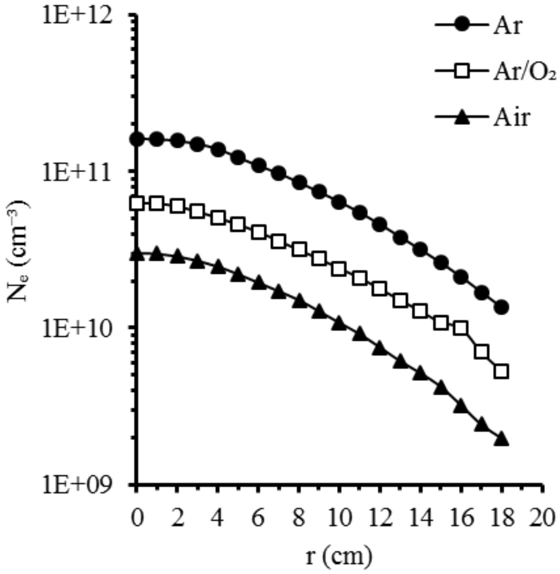

Fig. 4. Electron density measurements by Langmuir probe in plasma discharges filled by Air, Ar or Ar/O_2 (10%) mixture (15 Pa, 150 W).

The electron density shows a maximum value in the axis head source between 3.10^{10} and 2.10^{11} cm^{-3}, depending on the feed gas, followed by a decrease along the radial axis.

Microwave Interferometry (MWI) was used not only to compare the density obtained by Langmuir probe (LP) but also to achieve measurements closer to the source, at Z = 3.5 and 5.5 cm. These positions could not be reached with the probe.

This principle of MWI is based on the plasma-wave interaction. When the plasma is turned off, the microwave crosses the chamber without perturbation. When the plasma is switched on, the presence of electrons induces a phase shift of the microwave. This phase deviation is linked to the electron density by the following equation:

$$\langle n_e \rangle = \frac{n_c \lambda_0}{\pi L} |\Delta\varphi| \quad (2)$$

where n_c is the critical density, λ_0 the signal wavelength, L the plasma length and $\Delta\varphi$ the phase shift [5].

Fig. 5 represents the electron density as a function of radial position in the case of argon plasma (150 W, 15 Pa). It can be seen that closer to the source, the electron density reached almost 10^{12} cm^{-3}. At Z = 8.5 cm, a faster decrease of n_e is obtained by MWI. This is due to the difference of local and global measurements. Unlike probes which give local density values, interferometry method takes into account the density variation along the penetrated distance in the plasma by the microwave. Nevertheless, at r = 0 cm and Z = 8.5 cm, the density value is in perfect agreement.

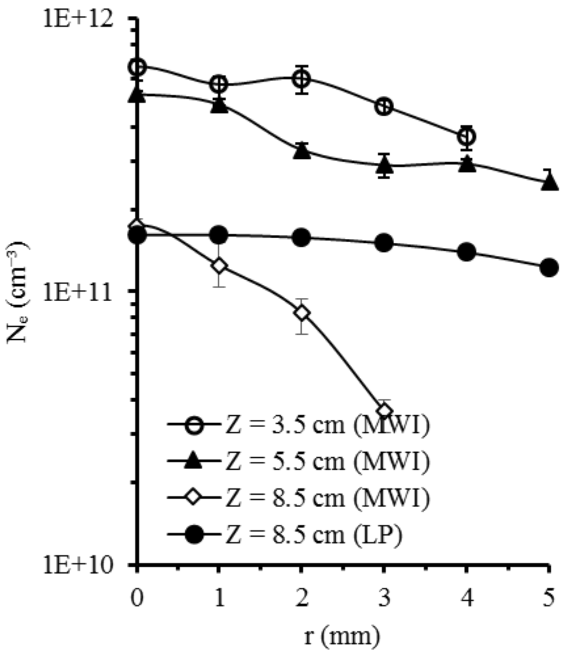

Fig. 5. Comparison of electron density measured by microwave interferometry (MWI) and Langmuir probe (LP) in argon plasma (15 Pa, 150 W) at different axial (Z) and radial (r) position.

3.3. Substrate temperature measurements

Two methods have been used to establish a relation between the microwave power injected in the discharge and the plasma particles contribution on the heating surface process [4].

The first method is based on the balance power provided by the microwave generator represented on Fig. 6. P_{MW} is the total power injected, considered mainly absorbed by the plasma with a small part dissipated through radiative processes and wall losses. P_{IN} is considered as the incoming power responsible of the substrate heating. P_S is the power deposited on the sample. Finally, P_{OUT} is the power released outside the substrate by radiation and thermal conduction.

P_{OUT} and P_S can be deduced through the temperature variation in the plasma-on/plasma off phase using the following equation:

$$P_{IN} = P_S + P_{OUT} = C_S\left(\frac{dT_H}{dt} - \frac{dT_C}{dt}\right)_{T_{Mean}} [W] \quad (3)$$

Where C_s is the caloric constant of the substrat, T_H the temperature measured in the heating phase, T_C the one measured in the cooling phase and T_{Mean} is the average temperature between the heating and cooling phases (see Fig. 7).

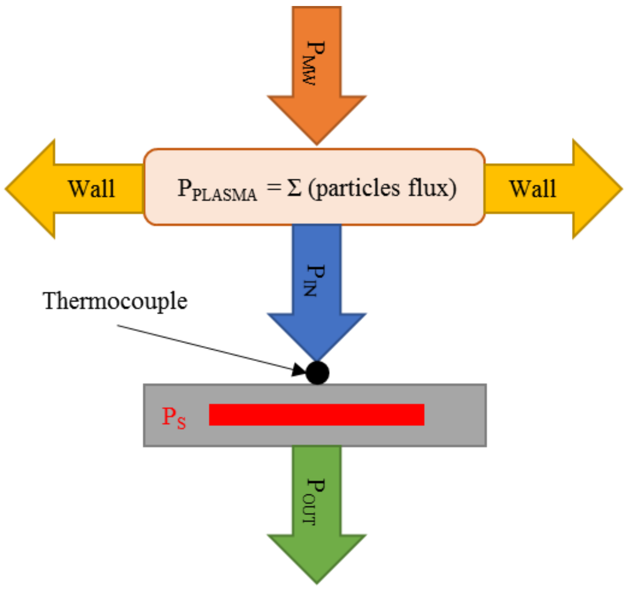

Fig. 6. Illustration of the power balance involved in the plasma-surface interaction.

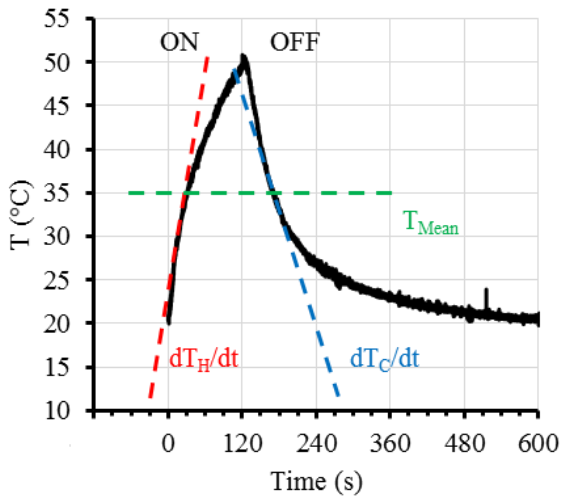

Fig. 7. Temperature variations measured by a thermocouple at the substrate surface.

Another possibility to calculate the total power influx at the substrate can be described by the sum of the particles and radiation contributions. In the case of low pressure microwave discharge, the main heating process is due to the particle bombardment [6]. It corresponds mainly to the electron-ion surface recombination, and the electron and ion flux reaching the surface.

Respectively, P_{recomb}, P_e and P_i in the following equation:

$$P_{PLASMA} = P_e + P_i + P_{recomb} \quad (4)$$

The complete formula of P_{recomb}, P_e and P_i are given by Kais et al [4].

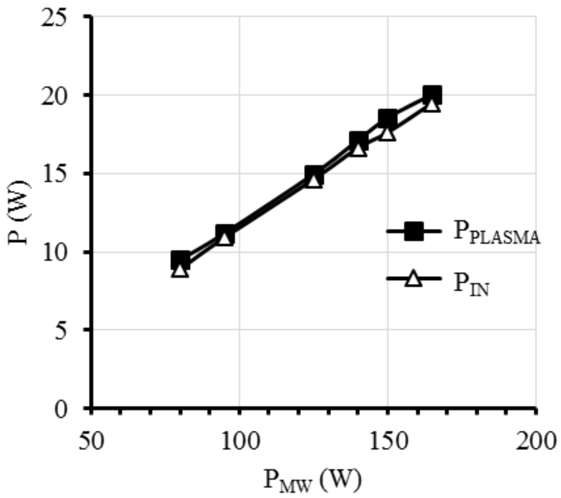

Fig. 8. Calculation of the microwave power transferred to the substrate surface.

Fig. 8 compares the results obtained by the two described methods. P_{IN} is calculated from the slope of the temperature variation. P_{PLASMA} is deduced by the particle contributions coming to the surface. First, this figures out a good agreement between P_{IN} and P_{PLASMA}. Then, a linear increase of P_{IN} with increasing the microwave power P_{MW} can be observed. Finally, the ratio P_{IN}/P_{MW} corresponds to the power efficiency. At 8.5 cm, 15 Pa and 150 W, 12.40% of P_{MW} is transferred to the substrate.

4. Conclusions

This study focused on the characterisation of a microwave plasma discharge. Optical emission spectroscopy has been used to estimate the electron temperature thanks to the Boltzmann plot method. The results have been compared and verified by Langmuir probe. A temperature varying between 1.3 and 2.0 eV depending on the pressure was measured in an argon plasma generated at 15 Pa and 150 W.

Langmuir probe and microwave interferometry was also applied to measure the electron density close to the sample in the discharge filled by Air, Ar or Ar/O_2 mixture gas. N_e varied from 3.10^{10} and 2.10^{11} cm^{-3} in the head source axis with a higher density in argon plasma. A decrease was observed along the radial axis up to a decade at r = 18 cm compared to the initial position (Z = 8.5 cm, r = 0 cm). Electron density close to the source could be achieved by microwave interferometry (Z = 3.5 and 5.5 cm). In the case of argon plasma, the electron density reached almost 10^{12} cm^{-3}.

The understanding of the heating sample process is important when the target can be thermo-sensitive. The quantity of microwave power P_{MW} transferred to the substrate has been established. The interpretation of the temperature variation curve or the calculation of the particle-surface interaction indicated that 12% of P_{MW} is deposited on the surface when the sample is positioned at 8.5 cm from the head source. This method could be extended to other plasma processes.

5. Acknowledgments

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6. References

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