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Autoreferát dizertačnej práce

**Štúdium kinetiky výbojov a zhášaných výbojov excitovaných
pri vysokých a veľmi vysokých frekvenciách v dusíkových zmesiach**

**Etude de la cinétique des décharges et post-décharges
excitées à hautes et très hautes fréquences dans les mélanges azotés**

na získanie akademického titulu philosophiae doctor

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Summary

Kinetic study of discharges and afterglows
excited at high and very high frequencies in nitrogen mixtures.

This work focuses on the study of the kinetics of discharges and post-discharges in flowing nitrogen and nitrogen mixtures at moderate pressures excited by radiofrequency and microwave cavities. The experimental diagnostic of these plasmas environments are mainly carried out by optical emission spectroscopy. Furthermore, an important work of kinetic modeling was performed on the basis of experimental results in order to understand the production and destruction processes of the species presents in the plasma. The thesis is divided into two parts according to the type of the discharge and gas mixtures studied.

In the frame of the part A, a N₂-Ar discharge generated at 27 MHz by an original helical cavity is examined by optical emission spectroscopy. The relatives intensities and characteristic temperatures of nitrogen species (N₂(B³Π_g), N₂(C³Π_u), N₂(a¹Π_g) and N(²P)) and those originated from impurities (OH(A²Σ⁺) and NO(A²Σ⁺)) are determined along with the vibrational distribution functions of the N₂(a¹Π_g, v' = 0 - 4) and N₂(C³Π_u, v' = 0 - 4) states. The discussion focuses on the evolution of species densities and temperatures as a function of the amount of argon in the mixture. The increase of this quantity resulted in (i) increased densities; (ii) high thermal imbalances except for the N₂(B³Π_g) state which remained a thermometric specie; and (iii) vibrational distributions with significant overpopulations which haven't been observed yet in the literature in the case of the state N₂(C³Π_u, v' = 0 - 4). Based on these experimental results, a global kinetic model coupled with a model of the vibrational distribution function of the N₂(C³Π_u, v' = 0 - 4) state have been developed. These models enable the analyze of our results and demonstrate the importance of the mechanisms involving metastable nitrogen and argon species, i.e. N₂(A³Σ_u⁺) et Ar(³P_{0,2}).

In Part B, discharges in mixtures of N₂-O₂ generated by a 433 MHz coaxial cavity were studied along with the afterglows. The methods of optical emission spectroscopy and mass spectrometry allowed to determine the evolution of concentrations of the main species produced (nitrogen atoms and oxides of nitrogen) based on the percentage of O₂ added directly to the zone of the discharge or that of the Lewis-Rayleigh afterglow. In order to describe the chemical processes taking place in the Lewis-Rayleigh afterglow, a time dependent 0D kinetic model was developed based on experimental data. The position of injection and the amount of O₂ strongly influence the formation of the studied species. This 0D kinetic model is associated with a flow simulation in order to qualitatively reproduce the evolution of concentrations and predict the nature of the environment created by the discharge as well as its characteristics for the applications of thin layer deposits of organosilicon materials for microtechnology.

Key words:

kinetic modeling, model of the vibrational distribution, time depending 0D kinetic model; nitrogen plasma, N₂-Ar, N₂-O₂; radiofrequency and microwave discharge; Lewis-Rayleigh afterglow; helical cavity; optical emission spectroscopy, mass spectrometry; metastable species.

Abstrakt

Štúdium kinetiky výbojov a zhášaných výbojov excitovaných pri vysokých a veľmi vysokých frekvenciách v dusíkových zmesiach.

Táto práca sa zameriava na štúdium kinetiky RF a mikrovlnných výbojov a zhášaných výbojov v dusíku a zmesiach plynov s dusíkom pri znížených tlakoch. Diagnostika spomenutých výbojov je realizovaná predovšetkým metódou optickej emisnej spektroskopie. Na základe experimentálnych výsledkov boli v predkladanej práci vyvinuté kinetické modely, ktoré predstavujú dôležité výstupy tejto práce. Cieľom výstupov je pochopiť procesy produkcie a zániku študovaných elektronických excitovaných stavov častíc prítomných v plazme. Práca je rozdelená na dve časti podľa typu výboja a študovaných zmesí plynov.

V časti A je študovaný výboj v zmesiach N_2 -Ar budený pri 27 MHz originálnym plazmovým zdrojom so špirálovou elektródou pomocou optickej emisnej spektroskopie. V tejto časti sú študované relatívne intenzity a charakteristické teploty excitovaných stavov dusíka ($N_2(B^3\Pi_g)$, $N_2(C^3\Pi_u)$, $N_2(a^1\Pi_g)$ a $N(^2P)$) a stavov molekúl resp. radikálov pochádzajúcich z nečistôt ($OH(A^2\Sigma^+)$ a $NO(A^2\Sigma^+)$). Spolu s intenzitami a teplotami sú študované aj vibračné distribučné funkcie molekúl $N_2(a^1\Pi_g, v' = 0 - 4)$ a $N_2(C^3\Pi_u, v' = 0 - 4)$. Diskusia je zameraná na vývoj koncentrácie a charakteristických teplôt molekúl v excitovaných stavov v závislosti od obsahu argónu v zmesi. Zvýšenie koncentrácie argónu zapríčiňuje (i) zvyšovanie koncentrácie stavov; (ii) odchýlky od termodynamickej rovnováhy prejavujúcej sa rozdielom rotačných teplôt molekulových stavov, okrem stavu $N_2(B^3\Pi_g)$, ktorého rotačná teplota je v najlepšej zhode s kinetickou teplotou plynu a teda umožňuje za týchto podmienok stanoviť teplotu plynu; (iii) výrazné odchýlky vo vibračnom rozdelení. Podľa dostupnej literatúry neboli vyššie spomenuté odchýlky vibračného rozdelenia $N_2(C^3\Pi_u, v' = 0 - 4)$ v takejto miere doposiaľ pozorované. Na základe experimentálnych výsledkov bol vypracovaný globálny kinetický model, ktorý je previazaný s modelom vibračného rozdelenia stavu $N_2(C^3\Pi_u, v' = 0 - 4)$. Spomínanými modelmi sme dokázali analyzovať výsledky a poukázať na dôležitosť procesov zahŕňajúcich metastabilné stavy ($N_2(A^3\Sigma_u^+)$ a $Ar(^3P_{0,2})$).

V rámci druhej časti sme študovali výboj a zhášaný výboj v zmesiach N_2 - O_2 budený mikrovlnným generátorom pri 433 MHz. Metódy optickej emisnej spektroskopie a hmotnostnej spektrometrie boli aplikované za účelom získať vývoj koncentrácií najdôležitejších molekulových stavov (dusíka a oxidov dusíka) v závislosti od obsahu kyslíka pridaného buď priamo do výboja alebo do priestoru zhášaného výboja (tzv. „dlho trvajúceho zhášaného výboja“). Za účelom popísať chemické procesy v „dlho trvajúcom zhášanom výboji“ bol na základe experimentálnych výsledkov vyvinutý časovo závislý 0D kinetický model. Pozícia pridania kyslíka má veľký vplyv na formáciu študovaných stavov. Tento kinetický 0D model je spojený so simuláciou prúdenia, čo nám umožňuje kvalitatívne reprodukovať vývoj koncentrácií excitovaných častíc a predpovedať charakter prostredia v prípade depozície tenkých vrstiev organokremičitých látok pre mikrotechnológiu.

Kľúčové slová:

kinetické modelovanie, model vibračnej distribúcie, časový 0D kinetický model; dusíková plazma, N_2 -Ar, N_2 - O_2 ; RF a mikrovlnný výboj; dlho trvajúci zhášaný výboj; plazmový zdroj so špirálovou elektródou; optická emisná spektroskopia, hmotnostná spektrometria; metastabilné stavy.

Introduction

The presented thesis is the result of an international collaboration in the frame of a PhD study under joint supervision between the „Department of Experimental Physics“ (Faculty of Mathematics, Physics and Informatics, Comenius University in Bratislava, Slovakia) and the „Plasma Processes and Materials Research Team“ (BioMEMS group at the Institute of Electronics, Microelectronics and Nanotechnology - UMR CNRS 8520 IEMN, University Lille 1, France).

The discharges and afterglows at low and moderate pressures (a few Pa - some hundreds of Pa) in mixtures containing nitrogen are frequently used in industry. Although at the present a large part of researches is focused on plasma technology at atmospheric pressure, the benefits of lower pressures continue to maintain this subject in the focus of many studies. For example, unlike the methods working at atmospheric pressure, it is possible to treat 3D objects uniformly, materials sensitive to oxidation or objects of larger dimensions. Additionally, the use of low pressure plasma technologies in the field of the manufacture of integrated circuits even after several decades remains a state of the art technology.

The generation of such a media is usually carried out by applying microwave or radio frequencies. This type of excitation combined with the reduced pressure leads to define the environment as “cold” plasma. Such environment contains electrons of significant energies, and ionized particles of lower energies which are in thermal equilibrium with the gas. Further, this medium includes metastable molecular species and vibrationally excited atoms which are significant energy vectors, even over long distances.

The thesis concerns the diagnostics of such an environment at moderated pressures in gas mixtures containing molecular nitrogen of various proportions. Through experimental and theoretical approaches it address two types of problems: (i) the understanding of plasma kinetics in N_2 -Ar mixtures of a RF discharge generated by an original plasma source, taking into account kinetic processes of N_2 molecules excited states with a state-to-state approach; (ii) the study of afterglows in microwave induced plasma of N_2 - O_2 mixtures (percentage of oxygen less than 25 %) as those implicated in processes of deposition of silicone polymer materials especially used in the laboratory IEMN for designing MEMS devices (MicroElectroMechanical Systems). On the basis of these subjects, the thesis is divided into two parts.

Part A

Part A of this work presents an experimental and theoretical study of a particular discharge. The originality is connected to how the plasma is excited. As a source a non commercial broadband helical cavity is used (see Figure 1). In the context of this study, the plasma is excited at a frequency of 27 MHz. However, the source is designed to operate in a wide frequency range from 13.56 to 2450 MHz. It is comparable to DC, RF and microwave sources, but offers the opportunity to work at different frequencies without changing the source itself. The excitation frequency of which depend the density and the electron energy distribution, and therefore the efficiency of ionization and dissociation, is important for the plasma media. The change in the excitation frequency implies a modification of

characteristic parameters of the plasma. Studies of its influence on the plasma-chemical processes, on the uniformity of species and of all plasma parameters are still among the actual research topics. The helical source was developed for this purpose.

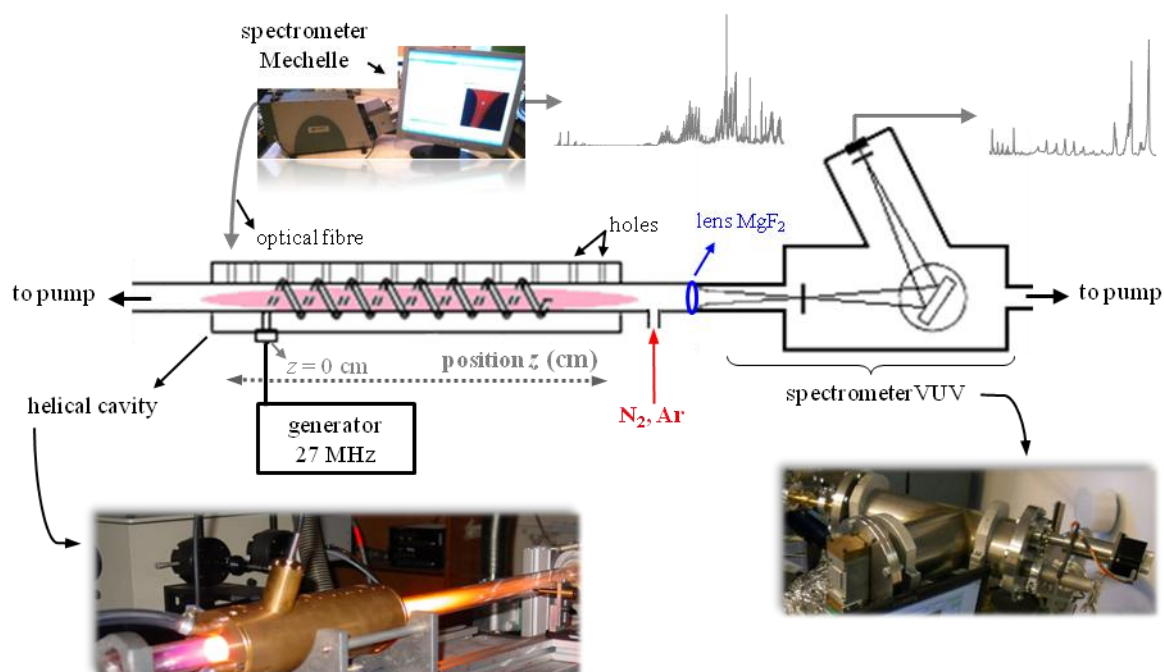


Figure 1: Experimental set-up from the part A.

Part B

Part B of this thesis concern the experimental and theoretical study of the Lewis-Rayleigh afterglow of N₂-O₂ mixtures formed downstream of a discharge N₂-O₂ at the pressure of several hundred Pa. We provide valuable analysis for fundamental research and also to understand the basic mechanisms for the control of processes related to surface modifications assisted by plasma. Indeed, the studied afterglow in this thesis is formed in a reactor intended to realize the deposition of thin films of silicone (see Figure 2 for the experimental set-up). The mixtures used for this type of application rely on nitrogen discharges producing afterglows with an eventual injection of O₂ and TMDSO (1.1.3.3.-TetraMethylDiSilOxane - reactive organosilicon precursor). The introduction of TMDSO leads to a ternary mixture which is much more complex with a kinetic that is poorly studied. In this context, firstly it is necessary to understand and control the plasma in N₂-O₂ mixtures and thus follow the kinetics of species (nitrogen and nitrogen oxides). Furthermore, optical spectroscopic investigations of an afterglow formed downstream of a discharge N₂-O₂ compared with studies of an afterglow with oxygen admixture formed downstream of a discharge of pure nitrogen give us the opportunity to analyze two different kinetics based on similar chemistry. The same species are indeed created but they are present in different proportions and have different ways of production schema. Thus we can conclude that for these different situations, the interactions between species in the gaseous medium and, ultimately, the substrate during the deposition process or surface modification will not be the same and will greatly influence the final properties of the material.

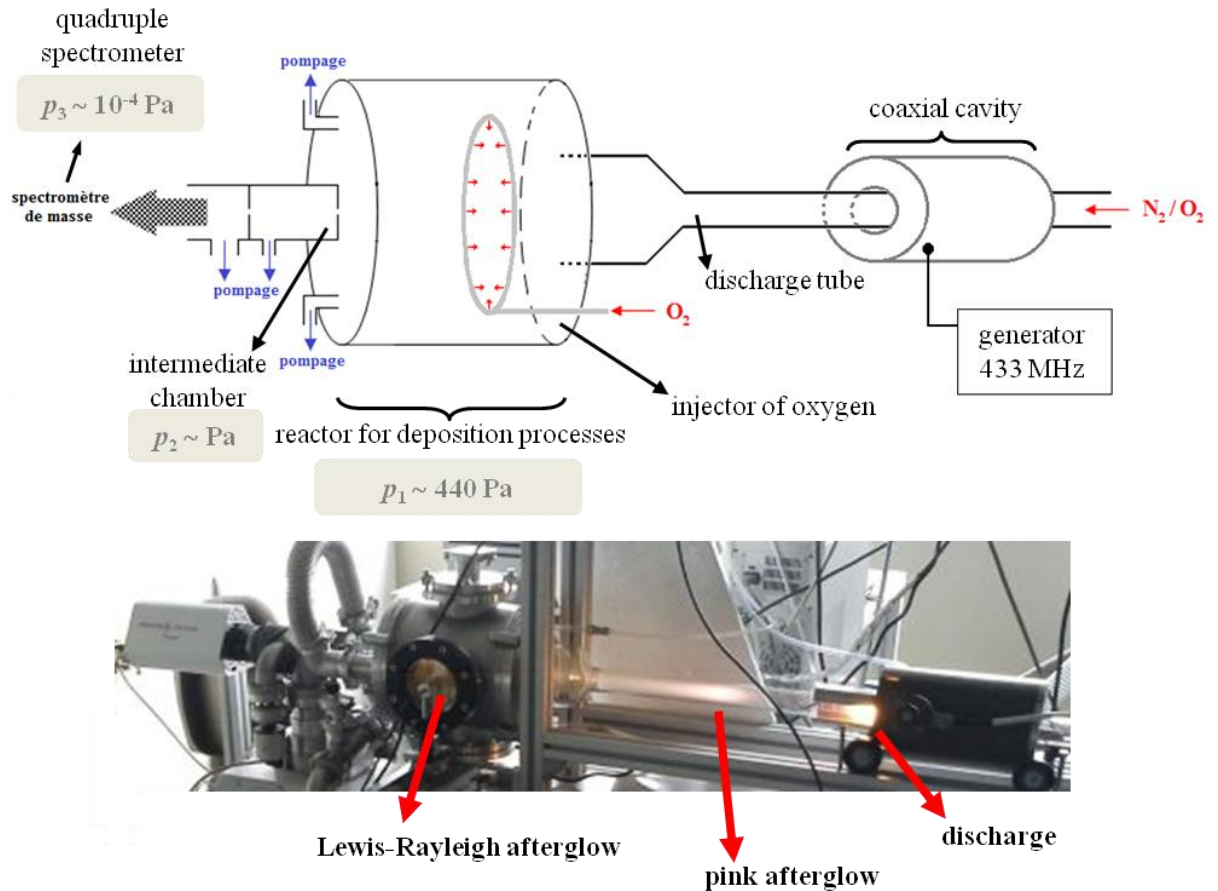


Figure 2: Experimental set-up from the part B.

Results

Part A was devoted to the study of the behavior of a RF discharge excited at 27 MHz by an original helical cavity in N_2 -Ar mixtures at moderate pressures (400 to 200 Pa) and low power (28 W).

As a first step, we conducted a study by optical emission spectroscopy spatially resolved in the spectral region from UV to near IR (135 - 950 nm). The main topic of this study concerns the effect of the argon concentration on densities and characteristic temperatures (gas temperatures, rotation and vibration) of the main species detected (nitrogen species: $N_2(a^1\Pi_g)$, $N_2(B^3\Pi_g)$, $N_2(C^3\Pi_u)$ and $N(^2P)$ and species from impurities: $OH(A^2\Sigma^+)$ and $NO(A^2\Sigma^+)$). The densities and temperatures were determined for different percentages of argon in the gaseous mixture (from 0 to 95 %) and different positions along the z axis of the cavity.

With regard to the determination of the characteristic temperatures, the technique of theoretical spectra simulations was used. Furthermore we have shown that the gas temperature was constant along the axis of the discharge equal to 430 ± 50 K for all our conditions studied. The densities of the studied species increased with the addition of argon. This increase is more pronounced for positions around

the center of the cavity. This spatial contrast is reduced by increasing the amount of argon in the gas mixture.

A second part of this study was focused on the identification and exploitation of the vibrational distribution functions of species $N_2(a^1\Pi_g, v' = 0 - 4)$ and $N_2(C^3\Pi_u, v' = 0 - 4)$ as function of the argon percentage and the position z along the discharge. The vibrational temperatures of these species can be determined only in the case of a discharge of pure nitrogen. In N_2 -Ar mixtures, overpopulations of the levels $v' = 2 - 4$ of $N_2(a^1\Pi_g, v')$ and the levels $v' = 1, 3$ and 4 of $N_2(C^3\Pi_u, v')$ appear progressively with the increase of the argon percentage.

As a third step, we developed kinetic models to analyze the experimental results. A global model for the evolution of species $N_2(B^3\Pi_g)$ and $N_2(C^3\Pi_u)$ based on their production and destruction mechanisms has been established. The system of coupled differential equations for the excited nitrogen molecules and atoms $Ar(^3P_{0,2})$ is solved numerically. This global model was then coupled with a second model describing the vibrational distribution function of the $N_2(C^3\Pi_u, v' = 0 - 4)$ state. The input parameters of the global model were the temperature of the gas, the electron density and temperature, vibrational temperatures of the $N_2(X^1\Sigma_g^+)$ and $N_2(A^3\Sigma_u^+)$ states and the rate of dissociation. The solution of the model provided the evolutions of the populations of the excited states $N_2(A^3\Sigma_u^+)$, $N_2(B^3\Pi_g)$, $N_2(C^3\Pi_u)$ and of the argon metastables, as well as the temperature and the electron density as function of the concentration of argon and the axial position z .

Hypothesis that explain the changes observed experimentally of the species $N_2(B^3\Pi_g)$ and $N_2(C^3\Pi_u)$ are discussed. This work shows in a clear and consistent way the roles of each energy vectors (electrons and metastable species $N_2(A^3\Sigma_u^+)$ and $Ar(^3P_{0,2})$) in the kinetics of the major emitters. The most important effects of the presence of argon are: (i) changes in the energy distribution function of the electrons with an increase in density and the temperature and (ii) the effectiveness of processes involving metastable nitrogen and argon species. These processes are primarily responsible for the increase in density of species $N_2(B^3\Pi_g)$ and $N_2(C^3\Pi_u)$ with the addition of argon in the mixture. The evolution of metastable species is a particularly interesting result of the model as it helps to understand the changes that occur in the rates of different production processes of the studied emitting states.

The complementary model on the vibrational levels of the $N_2(C^3\Pi_u)$ state allowed to adjust the input parameters of the global model, like the vibrational temperature of the $N_2(A^3\Sigma_u^+)$ state. The analysis of the vibrational distribution function of the species $N_2(C^3\Pi_u)$ also confirms the importance of metastable species in the kinetics of our discharge. Indeed, the phenomenon of strong overpopulation is due to an increase in terms of production related to the reaction of pooling involving two molecules $N_2(A^3\Sigma_u^+)$ particularly effective and pronounced in our discharge. Indeed, we show that this reaction is responsible for half of the production of $N_2(C^3\Pi_u)$ species. This specificity allows us to revisit the rate coefficients for the reaction of pooling. Besides, our analysis clearly shows that the model of Franck-Condon factors is not able to reproduce the experimental results. Only the change in the coefficients determined experimentally previously reproduces the structure of the vibrational distribution function observed by us.

This work, both experimental and theoretical, helped to better understand the original environment created by the helical cavity. This source had never been analyzed as in detail as here which led to a complete characterization of the kinetic. This work confirms the presence of metastable

species in large quantities which contributes to the excitation of species in the discharge. Furthermore, this study is also important in terms of fundamental research and completes the works on discharges in N₂-Ar mixtures at moderate pressures which were poorly studied so far.

Future studies of this discharge will aim to complement kinetic models by providing more experimental data, especially on the state N₂(A³Σ_u⁺) and the electron density and temperature. Furthermore, the validation of the models at low pressure is also required. It is also in perspectives to pass to another excitation frequency in order a further characterization of this original source and to investigate the influence of the excitation frequency on the plasma environment.

Part B was devoted to the study of flowing discharges and afterglows of N₂-O₂ mixtures excited at 433 MHz with a power of 250 W. The pressure and nitrogen flow are respectively 440 Pa and 1500 sccm. Oxygen was injected either to the discharge (flow rate from 0 to 500 sccm) or to the afterglow (flow rate from 0 to 200 sccm). The position of the addition of oxygen has been a major factor influencing the studied medium, the zone of the Lewis-Rayleigh afterglow formed in an organosilicon thin film reactor.

At first, we realized the experimental diagnostic of the afterglow by means of optical emission spectroscopy and mass spectrometry. These techniques allowed us to probe the nitrogen atoms in the ground state, N(⁴S), present in the Lewis-Rayleigh afterglow. In addition, the excited states of nitrogen and nitrogen oxides produced in the afterglow and discharge were examined by optical emission spectroscopy. The evolutions of the principal emission systems in the zone of the Lewis-Rayleigh afterglow originated from the molecules N₂(B³Π_g), NO(B²Π), NO(A²Σ⁺) were presented as function of the flow and position of the oxygen injection (either in the afterglow or in the discharge). Furthermore, in the case of O₂ injected in the discharge, we observed the emission of the continuum from the molecule NO₂(\tilde{A}^2B_2) as function of the O₂ flow rate.

Secondly, to determine the gas temperature, the rotational temperature of the N₂(B³Π_g) specie was determined. We observed an increase in the values of this temperature with the added oxygen flow in the discharge and afterglow, from 410 to 1400 ± 50 K and from 420 to 540 ± 50 K, respectively. These variations demonstrate the presence of disturbances observed in the afterglow originated from parasitic emission from the discharge that prevent to determine the temperature of the gas particularly in the case where O₂ is added in the discharge. Nevertheless, it is possible to determine the atomic density by quantifying the weight of the parasite transmission signal using the study of the vibrational distribution function of the N₂(B³Π_g, ν' = 2 - 12) specie. A methodology based on the assumption of a superposition of two signals, belonging to production mechanisms of N₂(B³Π_g, ν') state characteristics for the discharge zones (direct electron impact) and zone of the Lewis-Rayleigh afterglow (atomic three-body recombination) eliminates the signal parasite of the emissions from upstream of the studied areas. Comparison of results for the concentration of the N(⁴S) species obtained by the two diagnostic techniques shows a good agreement.

Then, based on the experimental results, a kinetic 0D model has been developed to account for the temporal changes in the densities of species: N(⁴S), O₂(X³Σ_g⁻), O(³P), NO(X²Π), NO₂(X), O₃(\tilde{X}^1A_1) and N₂(X¹Σ_g⁺, ν). The coupling with a stationary model involving some of these species has led to the theoretical calculation of the concentrations of NO(B²Π) and NO₂(\tilde{A}^2B_2). The results are

expressed as a function of flow rate of oxygen added in the discharge and in the afterglow. The two positions of O₂ injection offered the possibility to study the same species, but governed by slightly different kinetic schema. A quite satisfactory agreement between experiment and modeling was obtained. Finally, we have discovered strong bonds between the atomic species (N(⁴S) and O(³P)) and the molecules NO, NO₂ et O₃, which helped to identify the major processes enabling us to realize a simulation spatial-temporal of the medium despite the reactors complex geometry. For this purpose, we performed the simulation of the flow in our system using the COMSOL Multiphysics[®] software. The first attempts of calculations, with reasonable approximations to a set of constant reaction due to the reaction of O(³P) and NO(X²Π) gave very similar results, both qualitatively and quantitatively, as those provided by the 0D kinetic model. This recent but significant result allows considering the extension of the study to the case of the addition of O₂ in the afterglow of a nitrogen discharge, not treated in this work, but presenting an important step to effectuate the simulation of a reactor for deposition processes. Since the diagnosis of such a complex environment is essential for the control of industrial processes, we can conclude that we were able to provide elements of understanding of the kinetics of a N₂-O₂ afterglow which allow the transition to a study in the ternary mixture of N₂-O₂-TMDSO in the future.

This modeling, coupled with an experimental work in a medium for organosilicon thin films deposition establish a reference for the simulation of the environment of interest for the manufacture of MEMS in the laboratory of IEMN. Beyond the theme of deposition, the development of a tool for accurate simulation of afterglows of N₂-O₂ plasmas is show to be very important to see the distribution of species near of surfaces.

Conclusion

This thesis focused on the experimental and theoretical diagnostic of “cold” flowing plasma of nitrogen mixtures at moderate pressures. Two original studies were performed and detailed in parts A and B of the manuscript. One relates to a N₂-Ar plasma, the other to the afterglow of nitrogen plasma with or without O₂ added. These studies can be considered very actual and interesting from the point of view of industrial applications, but also in fundamental research. These two themes have a common point, because in general, few studies are devoted to theoretical diagnosis combines with experimental works, which is the originality of our study. It helps to provide answers as accurate and relevant as possible, because only the comparison with the experiment provides a validation of the theory.

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Štatistika kategórií (Záznamov spolu: 14):

ADC Vedecké práce v zahraničných karentovaných časopisoch (3)
AFC Publikované príspevky na zahraničných vedeckých konferenciách (6)
AFD Publikované príspevky na domácich vedeckých konferenciách (2)
AFG Abstrakty príspevkov zo zahraničných vedeckých konferencií (1)
AFH Abstrakty príspevkov z domácich vedeckých konferencií (1)
BEE Odborné práce v zahraničných zborníkoch (konferenčných aj nekonferenčných) (1)

Štatistika ohlasov (3):

[o1] Citácie v zahraničných publikáciách registrované v citačných indexoch (3)