

# Transmission of N-Atoms Produced by N<sub>2</sub> Flowing Microwave Afterglows through Hollow Tubes

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## ABSTRACT

Transmission of N-atoms ( $T_N$ ) through small diameters tubes (1.5 and 3 mm internal diameter (i.d) and 9, 50 and 80 cm length for silicone tubes, 1.5 mm i.d and 6.5 cm length for stainless steel tubes) has been measured in late N<sub>2</sub> and Ar-N<sub>2</sub> flowing afterglows of microwave plasmas in continuous and pulsed gas injection at a flow rate of 1 and 3 Standard liter by minute (Slm), a gas pressure from 2 to 4 Torr for N<sub>2</sub> and 20 Torr for Ar-1%N<sub>2</sub> and a plasma power from 150 to 300 Watt. From the experimental  $T_N$  values, it is deduced the  $\gamma$ -destruction probability inside the tube walls as being  $\gamma = (1-1.6) \times 10^{-3}$  for the silicon tubes and  $\gamma = (1.6-2) \times 10^{-2}$  for the stainless steel tubes.

**Keywords:** Spectroscopy; N-Atoms Transmission; Hollow Tubes;  $\gamma$ -Destruction Probability

## 1. Introduction

Production of N-atoms has been previously studied in flowing N<sub>2</sub> microwave post-discharges at medium gas pressures (1 - 10 Torr) [1]. Bacteria inactivation by N-atoms in such post-discharge reactors has been largely studied [1-3]. In these previous papers, it has been obtained a 6 log reduction of an initial bacteria population (*E. coli* 10<sup>6</sup> colony forming unit by milliliter, cfu/ml) deposited on planar surfaces with a 40 minutes exposure to a N<sub>2</sub> flowing afterglow at 350 K.

In the cited previous works, the N atoms are produced by microwave plasmas at power of 100 - 300 Watt. The plasma is located inside a quartz tube of internal diameter (i.d) 5 mm and extends along a few centimeters outside the cavity (surfatron) gap for a gas pressure between 1 and 30 Torr and a flow rate between 0.5 and 2 Slm.

In these conditions, the plasma gas temperature reached up to 1000 K near the surfatron gap [4], needing an air cooling of the quartz tube. After a few centimetres of post-discharge, it is found the room gas temperature.

After the plasma, early and late afterglows appear and the late afterglow extends in the post-discharge reactor where the sterilization of bacteria is studied.

It is presently reported the results obtained for the transmission of N-atoms produced in the N<sub>2</sub> late afterglow through small diameters hollow tubes.

These studies are of interest to appreciate the possibility of N-atoms sterilization inside hollow silicon and

stainless steel tubes.

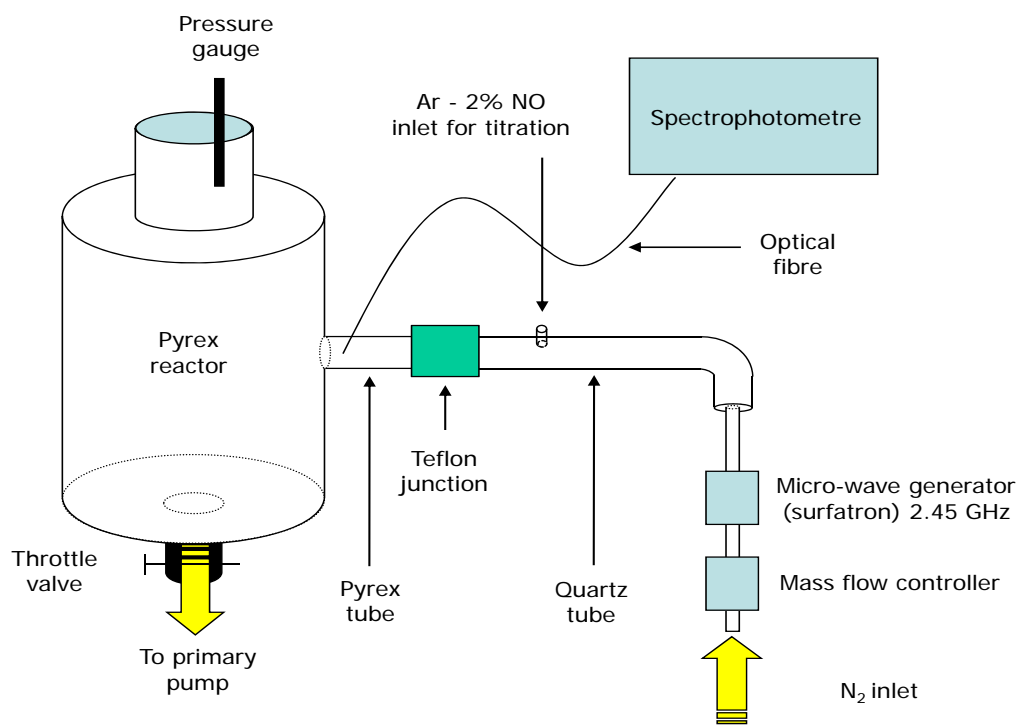
## 2. The Experimental Setup

The flowing microwave post-discharge reactor described in previous studies [1-3] is reproduced in **Figures 1(a)** and **(b)**. A two-stage oil rotary pump with a pumping speed of 48 sm<sup>3</sup>/h (800 slm) is set up after a 5 litre post-discharge reactor. The N<sub>2</sub> flow rate is controlled from 1 to 3 slm. The pressure inside the reactor can be adjusted by means of a throttle valve above the pump (see **Figure 1**).

A 2.45 Ghz microwave plasma is produced by a surfatron cavity in a quartz tube of 5 mm i.d and 30 cm in length. The discharge tube is connected to a tube of 18 mm i.d and 30 cm in length setup before the 5 litres post-discharge reactor in Pyrex glass. The dia.18 mm tube is in quartz after the dia.5 mm discharge tube and in Pyrex before the 5 litre reactor. A Teflon junction connects the two quartz and Pyrex tubes.

At a pressure of 5 Torr and a flow rate of 1 slm, the flow times are calculated as being  $2 \times 10^{-3}$  sec and  $10^{-2}$  sec in the tubes of 5 mm i.d and of 18 mm i.d., respectively.

The emission spectroscopy measurements are performed by moving an optical fibre along the tubes from the plasma gap and the external wall of the post-discharge reactor. The N atom density is obtained by NO titration [4] by injecting an Ar-2%NO gas mixture in the



(a)



(b)

**Figure 1.** Post discharge set up with the emplacement of the Teflon junction connecting the quartz and the Pyrex parts of the 18 mm i.d. tubes (a). The picture shows details of the Teflon junction and the emplacement of the three silicone tubes used in the transmission experiments (b).

post-discharge tube (see **Figure 1(a)**).

The transmission of N-atoms through hollow tubes was studied by using the Teflon junction allowing to join the quartz and Pyrex parts of the 18 mm i.d. tube **Figure 1(b)**.

Teflon junctions were worked to receive between 3 to 9 tubes of dia. 1 - 3 mm across it.

The insertion of hollow tubes in the Teflon junction produces pressure variations between the plasma tube and the 5 litre reactor.

Such pressure variations were analysed from the afterglow emissions in the 18 mm i.d tube at 2 cm before the Teflon junction (named hereafter  $z_0$ ) by keeping a given pressure in the 5 litre reactor.

The intensity  $I$  (580 nm) in  $z_0$  versus the gas pressure and for several flow rates is reproduced in **Figure 2** without the silicon tubes.

With the silicon tubes, the  $I$  (580 nm) intensity in  $z_0$  as reported in **Figure 2** allows to determine the upstream pressure for a given flow rate.

At a too high N<sub>2</sub> pressure ( $p > 5$  Torr), it was not always possible to switch on the N<sub>2</sub> plasma at a power less than 300 Watt.

Also as described in ref.5 it has been found that the plasma can be switched on at higher pressure by pulsing the gas. For that purpose, a Parker E.M valve was set up between the flow controller and the microwave cavity (see **Figure 1**)

In such a way the plasma was switched on as the gas pressure decreased when the valve was closed and it was maintained at high pressure when the valve was opened

(at a constant time of  $2 \times 10^{-2}$  sec).

An Ar-N<sub>2</sub> gas mixture was studied since the plasma is easier to sustain at high pressure [6].

### 3. Plasmas at High Gas Pressures

#### 3.1. Plasmas in Pulsed N<sub>2</sub> Gas

The microwave cavity was first tuned with a continuous gas inlet of 1 slm and an injected power between 100 and 300 Watt. Then by pulsing the gas with a  $\delta t$  pulse varying from 0.1 to 2 sec and a  $\Delta t$  period of 1 and 2 sec, it was successively produced two plasmas at high pressure (2 - 3 Torr) and at low pressure (0.4 - 0.5 Torr) [5].

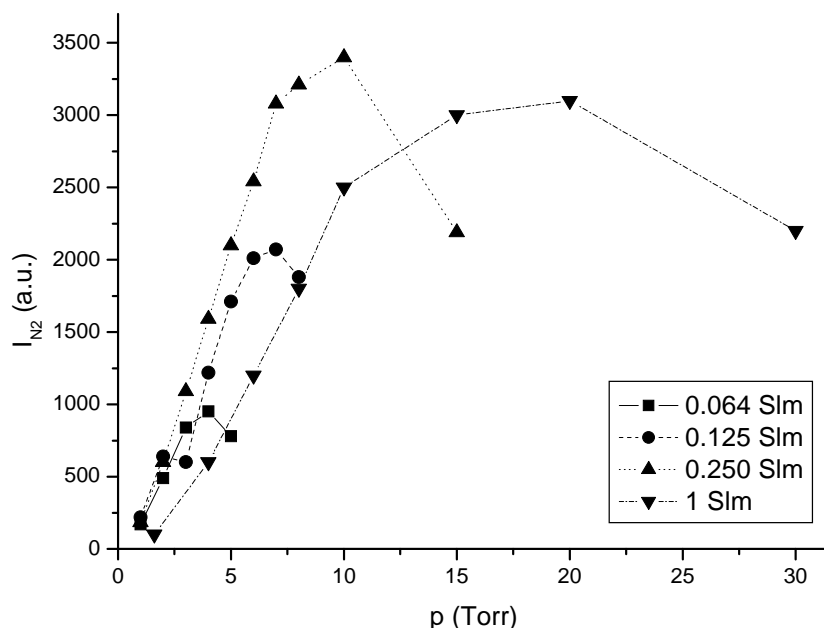
By checking the  $N_2^+ / N_2$  band intensity ratio near the surfatron gap, it was found that the microwave cavity tuning remained with the gas on, as for the continuous case with a negligible reflected power. At low pressure when the gas is off, the reflected power is important, varying from 30 to 90 Watt for an incident power from 100 to 150 Watt [5].

In these conditions, a strong late afterglow of N<sub>2</sub> 1st pos. bands (with a specific emission at 580 nm, resulting of N<sup>+</sup> N atoms recombination) was observed at high pressure and not at low pressure. As discussed in ref.4, the  $I(580 \text{ nm})$  afterglow intensity at high pressure ( $> 2$  Torr) is given by the following equation:

$$I(580\text{nm}) = k \cdot [N]^2, \quad (1)$$

where  $k$  is a constant.

By analysing the time varying gas pressure, afterglow intensity obtained with a photodiode (600 - 900 nm), it is



**Figure 2.** Variation of  $I(N_2, 580 \text{ nm})$  intensity in  $z_0$  with N<sub>2</sub> gas pressure without the silicon tubes, for several flow rates at 100 Watt.

obtained the results as reproduced in **Figure 3**.

It was established that for long pulses (0.4 - 1 sec), the N-atom density increased up to the end of gas pulse as did the plasma length

A maximum of N-atom density is obtained for time period  $\Delta t$ , twice the pulse time  $\delta t$ . The maximum pressure variations are for long pulses. In the following experimental conditions: incident power 200 Watt, flow rate 1 slm, pump speed 800 slm,  $\Delta t \sim 2\delta t = 2\text{sec}$ , it is determined a mean N-atom density of  $9 \times 10^{14} \text{ cm}^{-3}$  ( $10^{15} \text{ cm}^{-3}$  in continuous flow) and a pressure variation  $\Delta p = 2.3 - 0.5\text{Torr}$ .

### 3.2. Plasmas in Ar-N<sub>2</sub> Gas Mixtures

Homogeneous N<sub>2</sub> microwave post-discharges can be obtained in the continuous mode at reduced gas pressures (2 - 10 Torr). When the pressure is increased, the plasma became unstable and a 300 W power is not sufficient to sustain the discharge. Adding Ar to N<sub>2</sub> allows to obtain a stable discharge from low pressure up to atmospheric gas pressure with N<sub>2</sub> percentage below 23% with a microwave power limited to 300 Watt [6].

In the Ar-N<sub>2</sub> gas mixtures, the plasma gas temperature was found to be lower than 1000 K. In pure N<sub>2</sub> plasma, the electron energy distribution function is depleted above 3 eV because of the low mean vibrational excita-

tion of N<sub>2</sub> [7]. In contrast in Ar gas, the first excitation threshold being 11 eV, the extension of electron energy is higher. Consequently, by comparing to pure N<sub>2</sub> plasma the ionization in Ar-N<sub>2</sub> gas mixture is enhanced particularly at low N<sub>2</sub> percentage.

## 4. Transmission of N-Atoms in Hollow Tubes

### 4.1. Silicone Tubes

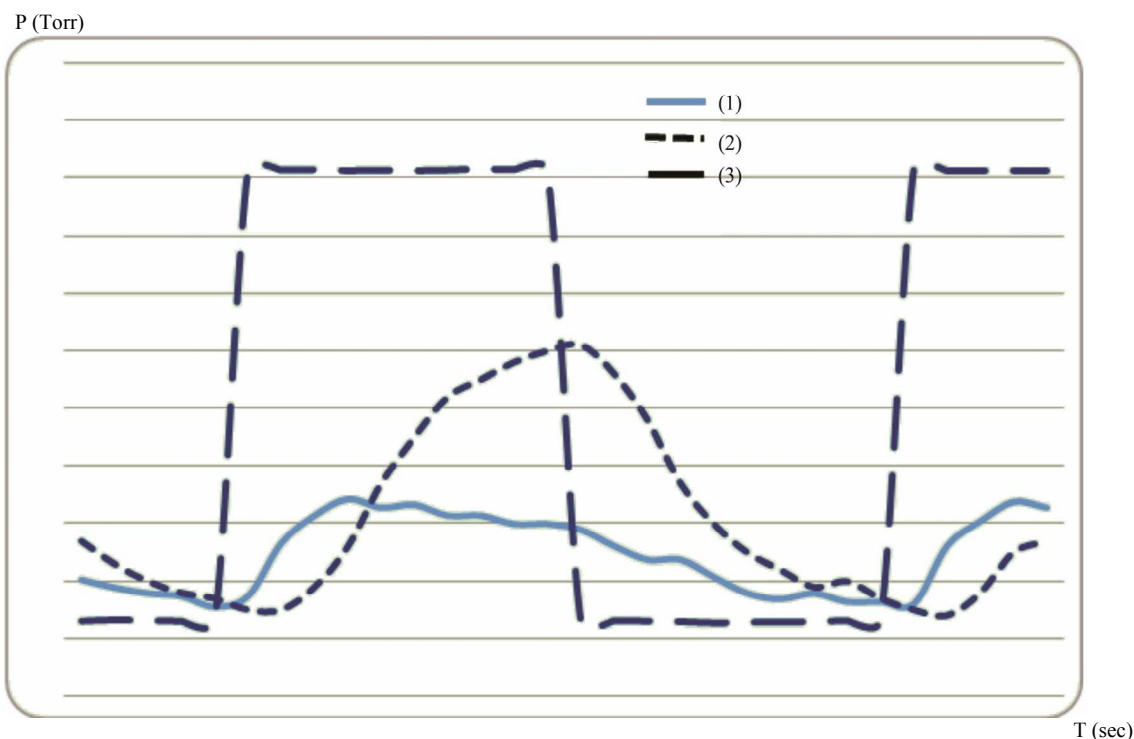
It was first experimented 9 tubes of silicones of int.dia. (i.d) 1.5 mm and out.dia. (o.d) 2 mm located inside the Teflon junction of **Figure 1** where 9 holes of dia. 2 mm were made inside a Teflon tap.

When the length of the tubes was 150 cm, it was necessary to pulse the gas with  $\Delta t \sim 2\delta t = 2\text{sec}$  at 200 Watt to switch on the N<sub>2</sub> plasma (see 3.1). In continuous mode, it was not possible to produce the N<sub>2</sub> plasma at 300 Watt.

In these pulsed conditions the afterglow with mainly emission of N<sub>2</sub> (580 nm) extended up to 50 cm inside the 9 tubes.

The length of the tubes was then reduced to 50 cm and the afterglow was detected inside the 5 litre reactor.

In conditions of  $\Delta t \sim 2\delta t = 2\text{sec}$ , 200 Watt, 1 slm, the gas pressure was found varying from 0.8 to 2.0 Torr in the 5 litre reactor and the N-atom density was obtained from the I(580 nm) intensity (see Equation (1)) after cali-



**Figure 3.** Variations with time of gas pressure (1) in Torr, of photodiode signal (2) and of electric field of the EM valve (3), obtained by the labview program. The photodiode and electric field signals are in arbitrary units. 150 Watt, 1 slm,  $\Delta t = 2\text{ sec}$ ,  $\delta t = 1\text{ sec}$ .

bration by NO titration [1-4].

It was found  $[N] = 3.4 \times 10^{15} \text{ cm}^{-3}$  at 1 cm before the Teflon junction. In the 5 litre reactor, the N-atom density was evaluated from the I (580 nm) intensity, corrected from the change of optical thickness between the Pyrex tube and reactor chamber. It was deduced  $[N] = 1.2 \times 10^{14} \text{ cm}^{-3}$  after 50 cm of silicon tubes length. The N-atom transmission  $T_N$  was thus evaluated to 3.5%.

A second experiment was undertaken with a Teflon tap bored of 3 holes of dia. 4 mm connecting 3 silicon tubes of i.d. 3 mm and o.d. 4 mm, of length from 80 to 250 cm.

For these larger diameter tubes, the N<sub>2</sub> plasma was switched on in the continuous mode.

At a flow rate of 1 Slm, a pressure of 4 Torr in the 5 litre reactor, a power of 150 Watt and silicon tube lengths of 80 cm, it is determined a N-atom density of  $2.7 \times 10^{15} \text{ cm}^{-3}$  before the Teflon tap and  $T_N = 8.5\%$  in the 5 litre reactor.

At a low flow rate of 125 Scm, a power of 100 Watt and silicon tube lengths of 60 cm, it was obtained a slow increase of  $T_N$  from 3.9% at 1 Torr to 4.9% at 3 Torr.

To improve the plasma lighting at high flow rate thus at high gas pressure, it was experimented as discussed in 3 - 2, an Ar-1%N<sub>2</sub> gas mixture.

Then the plasma was obtained in continuous mode at 150W, 3slm, 20 Torr and length 80 cm of the silicon tubes. The N-atom density before the Teflon tap was found to be  $8.2 \times 10^{15} \text{ cm}^{-3}$  and  $T_N = 8.5\%$  at 80 cm,

that is an increase of 3 of N-atom density and the same  $T_N$  value in comparison to N<sub>2</sub> 150 W, 1 slm.

## 4.2. Stainless Tubes

9 stainless tubes of i.d 1.5 mm and o.d 2 mm, of length 6.5 cm was located across the tap of the Teflon junction as for the silicon tubes in the 1st experiment in 4.1.

A continuous N<sub>2</sub> plasma was obtained at 300 W, 1 slm and 1.7 Torr in the 5 litre reactor. In these conditions, the N atom density was  $8 \times 10^{15} \text{ cm}^{-3}$  at  $z_0$ , before the Teflon junction and decreased sharply to about  $10^{14} \text{ cm}^{-3}$  at the exit of the 9 stainless tubes of length 6.5 cm, that is an N-atom transmission of 1.3%.

In the pulse mode  $\Delta t \sim 2\delta t = 2 \text{ sec}$ , 200 Watt, 1 slm, gas pressure varying from 0.9 to 2.3 Torr, the N-atom transmission was increased to 3.5% - 4%.

The  $T_N$  transmission through silicone tubes of int. dia. 1.5 and 3 mm and various lengths of 9, 10 and 80 cm in pulsed and continuous N<sub>2</sub> plasmas are reported in **Table 1(a)**. The result obtained with a continuous Ar-1%N<sub>2</sub> plasma is also reported. In **Table 1(b)**, it is reported the  $T_N$  values through stainless tubes of int.dia 1.5 mm and length 6.5 cm in pulsed and continuous N<sub>2</sub> plasmas.

## 4.3. $\gamma$ -Probability of N-Atom Destruction on the Tube Walls

By estimating that the main loss of N-atoms is by colli-

**Table 1. Transmission of N-atoms through: (a) Silicone tubes of int. dia. 1.5 and 3 mm and various lengths of 9, 10 and 80 cm. Pulsed and continuous N<sub>2</sub> plasmas. Continuous Ar-1%N<sub>2</sub> plasmas; (b) Stainless tubes of int.dia 1.5 mm and length 6.5 cm. Pulsed and continuous N<sub>2</sub> plasmas.**

(a) Silicone tubes									
Plasma gas	Number of tubes	Tube i.d. (mm)	Operating mode	Power (W)	Flow rate (slpm)	Pressure (Torr)	$[N]_{z_0}$ ( $10^{15} \text{ cm}^{-3}$ )	Tube length (cm)	$T_N$ (%)
N <sub>2</sub>	9	1.5	Pulsed $\Delta t \approx 2\delta t = 2s$	200	1.0	0.9 - 2.3	3.5	10.0	30.0
N <sub>2</sub>	9	1.5	Pulsed $\Delta t \approx 2\delta t = 2s$	200	1.0	0.9 - 2.3	3.5	50.0	3.0
N <sub>2</sub>	3	3.0	Continuous	300	1.0	4.0	4.2	9.0	35.0
N <sub>2</sub>	3	3.0	Continuous	300	1.0	4.0	4.2	80.0	8.5
Ar-1% N <sub>2</sub>	3	3.0	Continuous	150	3.0	20.0	8.2	80.0	8.5
(b) Stainless steel tubes									
Plasma gas	Number of tubes	Tube i.d. (mm)	Operating mode	Power (W)	Flow rate (slpm)	Pressure (Torr)	$[N]_{z_0}$ ( $10^{15} \text{ cm}^{-3}$ )	Tube length (cm)	$T_N$ (%)
N <sub>2</sub>	9	1.5	Pulsed $\Delta t \approx 2\delta t = 2s$	200	1.0	0.9 - 2.3	8.3	6.5	3.5 - 4.0
N <sub>2</sub>	9	1.5	Continuous	300	1.0	1.7	8.0	6.5	1.3

sions on tube wall, it is written:

$$[N]_z = [N]_0 \cdot \exp\left[-\gamma(c'/2R)(z/v)\right] \quad (2)$$

where  $z$  is the axial distance in the silicon tube,  $[N]_0$  is the N-atom density at  $z_0$ ,  $c'$  is the thermal gas velocity at 300K:  $c' = 5 \times 10^4 \text{ cm s}^{-1}$ ,  $R$  is the tube radius and  $v$  the axial gas velocity.

The axial gas velocity is deduced from the flow rate  $Q$  by:

$$Q(p) = S \cdot v \quad (3)$$

where  $Q(p)$  is the flow rate at pressure  $p$  (Torr):  $Q(p) = Q_0 760/p$ ,  $Q_0$  is the standard flow rate (760 Torr, 300K) and  $S$  is the tube internal surface.

To obtain a nearly constant gas pressure in the 3 silicon tubes, the flow rate was decreased to 0.125 Slm for a pressure of 1 - 3 Torr in the 5 litre reactor.

At  $Q = 0.125 \text{ Slm}$ , the I (580 nm) measured in  $z_0$  with the silicon tubes at 2 Torr indicates a pressure before the Teflon junction of 2 - 3 Torr.

With Equation (3), it is calculated at 2 Torr an axial gas velocity of  $3.7 \times 10^3 \text{ cm} \cdot \text{sec}^{-1}$  corresponding to a flow time in the silicon tubes of  $1.6 \times 10^{-2} \text{ s}$  at 60 cm.

Then, it is determined from Equations (2) and (3), a  $\gamma$  value varying from 1.2 to  $0.8 \times 10^{-3}$  as the pressure in the reactor increased from 1 to 3 Torr.

Such values are 3 times lower than published values for Nylon [8]:  $\gamma = 3 - 2 \times 10^{-3}$  at 2 - 3 Torr.

The  $\gamma$ -value has been calculated with the Ar-1%N<sub>2</sub> gas mixture in conditions of 3 slm and 20 Torr in the 5 litre reactor. From the I(580 nm) intensity calibrated with the Ar-1%N<sub>2</sub> gas mixture as for N<sub>2</sub> in **Figure 2**, it is estimated a pressure of 45 Torr in front the Teflon tape. By taking the mean value of pressure  $p_m = 32$  Torr in the tubes, it is calculated from Equation (3) an axial velocity of  $5.6 \times 10^3 \text{ cm} \cdot \text{s}^{-1}$ . At 80 cm, it is measured  $T_N = 8.5\%$ . It is then calculated from Equation (1) that  $\gamma = 10^{-3}$ .

In N<sub>2</sub>, 1 slm, 150 Watt, the pressure before the Teflon tape is found from **Figure 2** to be 10 Torr. For a pressure of 4 Torr in the 5 litre reactor, the mean pressure in the tubes is 7 Torr, the axial velocity is  $8 \times 10^3 \text{ cm} \cdot \text{s}^{-1}$ . With  $T_N = 8.5\%$  at 80 cm, it is deduced  $\gamma = 1.6 \times 10^{-3}$  near the published value for Nylon at 4 Torr:  $\gamma = 2 \times 10^{-3}$  [8].

It is concluded to about the same value of  $\gamma$  probability of N atoms in pure N<sub>2</sub> and in the Ar-1%N<sub>2</sub> gas mixture:  $\gamma = (1 - 1.6) \times 10^{-3}$ . The argon gas has no influence on the N-atom destruction probability on the silicon tubes walls.

With such  $\gamma$  values, it can be calculated the N-atom density at the exit of the 3 silicon tubes of i.d. 3 mm and length 250 cm:  $1.3 \times 10^{12} \text{ cm}^{-3}$  for N<sub>2</sub> at 1 Slm, 150 W and 4 Torr in the 5 litre reactor and  $5.7 \times 10^{12} \text{ cm}^{-3}$  for Ar-1% N<sub>2</sub> at 3 Slm, 150 W and 20 Torr. The increase of N-atoms density by 4.4 when N<sub>2</sub> is changed into the Ar-1%N<sub>2</sub> gas mixture comes from the increases by a fac-

tor 3 of N-atom density before the Teflon tape and by a factor 3 of flow rates in spite of increase of pressure in the tubes of length 250 cm from about 20 Torr in N<sub>2</sub> to 30 Torr in Ar-1%N<sub>2</sub>.

Equations (2) and (3) are applied to the results of the stainless tubes in part 4.2.

The pressure before the tap of the 9 stainless tubes was determined from the I (580 nm) intensity as it has been experimented with the silicon tubes. It is estimated a pressure of 10 - 16 Torr for 4 Torr in the 5 litre reactor. The mean pressure in the tubes is taken between 7 - 10 Torr.

With a N-atom transmission of 1.3% in continuous mode after 6.5 cm of tube length, it is calculated  $\gamma = (1.6 - 2) \times 10^{-2}$ .

This value is 3 - 4 times higher than the published values in pulsed RF discharge for stainless steel [9]:  $\gamma = 5 \times 10^{-3}$  at 5 Torr and  $\gamma = 6 \times 10^{-3}$  at 3 Torr.

## 5. Conclusions

Transmission of N-atoms through hollow tubes in the late flowing afterglow of a N<sub>2</sub> microwave discharge has been measured for silicon tubes of i.d. 3 mm and a length up to 250 cm and for stainless steel tubes of i.d. 1.5 mm and 6.5 cm length. The hollow tubes were placed though holes in a Teflon tap across a Teflon junction of i.d. 18 mm in the main N<sub>2</sub> flow before a 5 litre reactor. Depending on the gas flow, a gas pressure variation is produced before and after the Teflon junction. To switch on the plasma at high gas pressure, it was experimented pulsed N<sub>2</sub> gas modes and Ar-N<sub>2</sub> gas mixtures. The pulse gas mode was of interest to produce the plasma at lower gas pressure when the gas was off and to maintain it at high pressure when the gas was on.

In comparison to continuous discharges, the N-atom transmission across the 1.5 mm i.d stainless steel tubes increased with the pulsed gas mode.

An Ar-1%N<sub>2</sub> gas mixture has allowed to produce plasmas at high flow rates and gas pressures with higher N-atom density than in pure N<sub>2</sub>.

The  $\gamma$ -destruction probability has been determined inside the tube walls as being  $\gamma = (1 - 1.6)$

$10^{-3}$  for the silicon tubes and  $\gamma = (1.6 - 2) \times 10^{-2}$  for the stainless steel tubes. The  $\gamma$ -values are in good agreement with published values for the silicon tubes but not for the stainless steel tubes which appear to be 3 - 4 times higher.

With a value  $\gamma = 10^{-3}$ , the best result for N-atom injection in the silicon tubes of 3 mm i.d and length 250 cm is obtained with an Ar-1%N<sub>2</sub> gas mixture at 3 Slm, 150 W giving a measured density of  $8 \times 10^{15} \text{ cm}^{-3}$  before and a calculated density of  $6 \times 10^{12} \text{ cm}^{-3}$  after the tubes in the 5 litre reactor.

## 6. Acknowledgments

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