Vibrational Temperature Estimation of Nitrogen Molecules in Radio-Frequency (RF) Produced Plasma^{*)}

Nandini YADAVA^{1,2)}, Sachin S. CHOUHAN³⁾, Amulya SANYASI⁴⁾, Uttam SHARMA³⁾, Jayashree SHARMA³⁾, Malay B. CHOWDHURI⁴⁾, Joydeep GHOSH⁴⁾ and Ankur PANDYA⁵⁾

¹⁾Institute of Science, Nirma University, Ahmedabad 382 481, Gujarat, India

²⁾Department of Aerospace Engineering, Indian Institute of Technology Kanpur, Kanpur 208 016, Uttar Pradesh, India

³⁾Shri Vaishnav Vidhyapeeth Vishwavidyalaya, Sanwer Road, Indore 453 111, India

⁴⁾Institute for Plasma Research, Bhat, Gandhinagar 382 428, India

⁵⁾Institute of Technology, Nirma University, Ahmedabad 382 481, Gujarat, India

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A capacitive coupled radio frequency (RF) plasma system has been developed for producing tungsten coated graphite tiles using plasma assisted chemical vapor deposition (PACVD) technique. To characterize the deposition chamber for optimal plasma parameters, small amount of air is released into the hydrogen plasma purpose-fully to measure its gas temperature using spectral bands of nitrogen molecule. Optical emission spectra in the wavelength range 350 to 900 nm have been recorded with a miniature spectrometer. Molecular spectral bands of N₂ (B³ \prod_g -A³ \sum_u^+) have been observed and identified as three bands from the nitrogen 1PS ($\Delta v = 2$, 3 & 4). These bands are simulated using MATLAB code developed in-house by considering Boltzmann distribution of particles in the vibrational states. The experimental spectra have been modelled with the simulated spectrum through the best-fit technique by iterating the latter one with different temperature values. Boltzmann plot method is also utilized to evaluate plasma gas temperature using identified vibrational bands. The estimated temperature using spectral modelling method matches fairly well with Boltzmann plot method. The estimated vibrational temperature are in the range of ~7000 - 8000 K, an order higher than the room temperature ~300 K.

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1. Introduction

Plasma based technologies are increasingly used for coatings in numerous applications ranging from optics, automotive, aerospace, biomedical, fusion and other R&D areas. Radio-frequency (RF) generated capacitive coupled plasmas are advantageous over other methods used in the plasma-assisted material processing. RF capacitive coupled plasmas are low cost, large area interaction and high repeatability over other methods [1]. The 13.6 MHz frequency is preeminent method for low pressure capacitive (parallel plate) RF discharges. In this frequency range, electrons gain only few eV of energy to decompose the molecules. Furthermore, the electron density is also low and therefore the amount of heat transfer due to the electron is also small which facilitates the coating (contact) with sensitive surfaces [2]. This coating is processed with different chemical vapor deposition (CVD) techniques. One among them is the plasma assisted chemical vapor deposition (PACVD) [3]. In fusion grade machines such as tokamak, plasma facing compo-

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nents (PFC) plays an important role for better confinement of the plasmas. The fusion grade material requires to have low fuel retention with high heat load handling capabilities and low sputtering yields. This produces clear demand on the choice of materials of PFCs [4]. These issues can be overcome by the use of tungsten coated graphite tiles [2]. Worldwide different techniques have been developed and attempted for tungsten coatings on the different material surfaces. ADITYA-U is a medium sized tokamak having major radius 75 cm and minor radius 25 cm with toroidal magnetic field of 0.75 - 1.5 T at the Institute for Plasma Research (IPR), India [5]. It confines the high temperature plasma in the limiter as well as divertor configurations. The graphite limiter or divertor tiles of ADITYA-U tokamak are planned to be coated with tungsten using PACVD technique in the reactor [6]. For this purpose, at the Shri Vaishnav Institute of Technology and Science (SVITS), Indore a coating reactor based upon capacitively coupled RF plasma is fabricated for producing tungsten coated graphite tiles using PACVD technique to be tested as a part of first wall components in ADITYA-U tokamak [6,7].

Beside the measurement of basic plasma parameters (plasma density, electron temperature, and ion energy) in

author's e-mail: nandini7754@gmial.com

PACVD, it is important to estimate the plasma gas temperature, T_g to understand the reaction chemistry. The gas temperature influences the rate coefficients of active species generation through the different molecular processes. These processes are mainly dissociation, excitation, and ionization. The properties of deposited film or material, such as its uniformity along with certain intrinsic properties, are also influenced by the gas temperature gradients present in the closeness of the substrate. Moreover, T_g is strongly linked with the density of the heavy species. Therefore, accurate information on the gas temperature is required to understand and control plasma processes.

Optical emission spectroscopy (OES) is the most popular way to determine the plasma gas temperature. To use OES, it is required to identify the population model (energy distributions of particles), which is important to calculate the basic plasma gas temperature. Population models are decided by the plasma density, temperature and applied pressure in the plasma device. Typically, high pressure discharges lead to high density plasma that happens to be in the local thermal equilibrium (LTE). However low pressure and low-density plasmas are far from LTE, the model employed for such plasma is non-LTE or corona model. OES measurements using rovibrational band spectrum are good indicators to the overall plasma gas temperature. By adding additional amount of N2 into the plasma, one can observe the nitrogen band structures. This can be used as a sensitive "thermometer" for the gas temperature measurement, i.e., a molecular actinometer. The vibrational temperatures are usually estimated by fitting Boltzmann distribution. The plasma gas temperature can differ strongly from the electron temperature in plasmas because the molecular population processes is different from the electron collision process. Vibrational temperature can also provide insights about the relative rates of vibrationvibration and vibration-translation energy exchange processes

In this study, the experiment is performed in a PACVD reactor having plasma density in the range of $\sim 10^{16} \text{ m}^{-3}$ and electron temperature $\sim 1 \text{ eV}$ [6]. The visible spectra are obtained by a spectrometer having spectral resolution of 0.2 nm. Based on spectral line identification, three bands from nitrogen first positive system are identified B³ Π_{g} -A³ Σ_{u}^{+} , the band having $\Delta v = 2$ present at 710 to 790 nm, $\Delta v = 3$ present at 620 to 690 nm and $\Delta v = 4$ present at 560 to 620 nm.

A MATLAB based code has been developed to simulate the theoretical bands for different temperatures, at certain temperature the experimental and simulated bands are matched with each other. This temperature is also matched with the gas temperature calculated by Boltzmann plot method.

The paper is organized as follows. In Section 2, we briefly discuss about the experimental setup. Section 3 presents the techniques for Boltzmann plot and spectral band simulation and the results on the plasma gas temper-

ature measurement. The effect of Fulcher- α band on the proper estimation of gas temperature by both the measurement techniques are discussed in Section 4.

2. Experimental Setup

The main purpose of the experimental setup is to produce a reactor for tungsten coating experiments, which has application in the tokamaks. For this purpose, the cylindrical shaped vacuum vessel is developed which has 360 mm of diameter and 300 mm of height, made-up of Stainless Steel (SS 304). It has five 35 CF viewports mounted on top and eight 63 CF viewport placed radially to arrange different, diagnostic, pumping system, gauge, probe, etc. The glow discharge plasma is produced in a chamber having two circular metallic disc electrodes having diameter 100 mm and spacing of 25 mm which are powered by RF source having frequency 13.6 MHz and power 600 W. The upper electrode is powered, and the lower is grounded. The schematic of experimental setup is shown in Fig. 1. The system is initially brought to the roughing vacuum with rotary pump of capacity 12 m³/hr, then turbo-molecular pump, which has capacity of 60 liters/sec, is used on the system to achieve the base pressure. The vacuum is monitored by Pirani and Cold cathode gauges. Generally, for the tungsten coating experiments, tungsten hexafluoride (WF₆) is used as precursor gas and inserted in to the background hydrogen plasma. In this study prior to feeding the hydrogen gas, the system is pumped down to 2×10^{-5} mbar and hydrogen discharge are produced with hydrogen fill pressure $\sim 5 - 8 \times 10^{-3}$ mbar with 80 W of RF power.

The optical emission spectroscopy measurement is carried out and setup is shown in Fig. 1. The spectroscopic setup is arranged on the 63 CF radial view port. The optical window is made of fused silica and sufficient light is transmitted to the spectrometer with light collecting optics. Light collection optics includes collimating lens and 1 mm core diameter optical fiber. The light is then transmitted to the entrance slit of the spectrometer. Spectrometer used during experiment is a compact high per-



Fig. 1 Schematic of experimental setup.

formance, miniature spectrometer (Make: Ocean optics; USB4000). This comes with standard 3648-pixel, builtin detector (Make: Toshiba; TCD1304AP). It is capable to cover visible wavelength range \sim 350 - 900 nm with 300 grooves/mm grating. This grating is blazed at 650 nm. The spectrometer is powered and controlled by the USB connected to the computer and company made licensed software, Spectra Suit. It has fixed slit width of 20 um and exposure time can be changed. During experimental results discussed here, the exposure was kept at 1 sec. The wavelength calibration has been carried out using pen ray lamp source (UVP made), with different mercury lines. The line of sight views the discharge inside the deposition chamber along a chord passing through the central mid-plane of the plasma as shown in Fig. 1. The data presented here are obtained after the plasma is stabilized inside the chamber.

3. Measurement Technique

The vibrational temperature (T_{VE}) of the molecule present in the plasma is the representation of the plasma gas temperature (T_g). N₂ molecules give vast number of molecular bands in the visible range, which is easy to detect. Then the characterization of plasma gas temperature becomes easy by feeding small amount of nitrogen gas in hydrogen produced plasma. The nitrogen (air) is then leaked into the stable hydrogen plasma while continuously monitoring the spectral emission from the plasma. The nitrogen (air) leak is maintained at a level where N₂ molecular lines with sufficient spectral intensity is observed.

The most common Boltzmann plot method is routinely used to determine the electronic, vibrational and rotational temperatures in the plasmas [8]. This concept has been utilized here to estimate the vibrational temperatures from the measured spectra of N_2 vibrational bands. This technique is mentioned here as the experimental estimation of temperature. The complete idea of diatomic molecular spectral and their terms may be found in references [9,10].

The intensity of the spontaneous emission radiated by excited molecules from vibrational state of upper level to the vibration sate of lower level is given by the following equation [11, 12].

$$I_{v'v''} = \frac{hv_{v'v''}}{4\pi} g_v A_{v'v''} n_0 e^{-\frac{E_{v'}}{k_B T_{VF}}}.$$
 (1)

Here v' and v'' stand for vibrational quantum number of the upper and lower states respectively. $I_{v'v''}$ is the measured intensity of the transition, $A_{v'v''}$ is the Einstein coefficient, g_v is the statistical weight of the vibrational level of the molecule, T_{VE} is the vibrational temperature, k_B is the Boltzmann constant and $E_{v'}$ is the total vibrational energy of molecules. The vibrational energy for a level v' is described as an anharmonic oscillator

$$E_{v'} = w_e \left(v' + \frac{1}{2} \right) - w_e x_e \left(v' + \frac{1}{2} \right)^2.$$
 (2)

Where, ω_e is the vibration constant, and χ_e is the anharmonicity constant. Anharmonicity effects are neglected in the present computational model [13].

Further simplifying the equation, we have:

$$\ln\left(\frac{I_{v'v''}\lambda_{v'v''}}{g_{v}A_{v'v''}}\right) = -\frac{E_{v'}}{k_{B}T_{VE}} + C.$$
(3)

The slope (m) of the above equation $m = -\frac{1}{k_B T_{VE}}$ gives the vibrational temperature, T_{ve} , which is obtained by plotting $\ln\left(\frac{I_{v'v'}\lambda_{v'v'}}{g_v A_{v'v'}}\right)$ versus $E_{v'}$. Where $g_v = 1$ for the vibrational levels v of the diatomic molecules.

Another measurement technique, mentioned here as the simulated estimation of temperature, is based upon simulation or modelling the spectral line shape profiles of the vibrational bands considering the same intensity distribution represented in equation (1). The spectral line is considered to be a Gaussian line shape profile function $P(\lambda, \lambda_{FWHM})$, given by the following equation:

$$P(\lambda, \lambda_{FWHM}) = \sqrt{\frac{4\ln 2}{\pi}} \frac{1}{\lambda_{FWHM}} \exp\left[-4\ln 2\left(\frac{\lambda - \lambda_0}{\lambda_{FWHM}}\right)^2\right].$$
 (4)

Here, λ_{FWHM} is the full width at half maximum (FWHM) of the Gaussian profile. The final modelled intensity of a single molecular transition depends upon FWHM of the line shape; however, its intensity is variable parameter depending upon the temperature value as given in equation (1). Thus, by changing FWHM and temperature, at a certain temperature value, the experimental spectra match with the simulated or modeled spectra. The results from direct experimental measurement and simulated spectra measurement are then compared further. The technique involving simulation of the spectra is more advantageous as in the experimental measurement technique, fitting error is introduced twice; one is during multi-peak Gaussian fitting and another during linear fitting of Boltzmann plot. However, in the simulation-based measurement, proper iteration would provide better temperature measurement since only Gaussian fitting error now comes into the estimation procedure.

4. Result on the Gas Temperature Measurement

The observed spectrum is shown in Fig. 2. This emission spectrum is dominated by N₂ molecular bands from the 1st positive system (1PS), which mainly falls between 550 to 800 nm wavelength range, it is identified as N2 1PS. This corresponds to the transition $B^3\prod_g -A^3\sum_u^+$ where three band transitions are identified with $\Delta v = 2$, 3, and 4. Second positive system of nitrogen (C-B) is also present in the same spectrum and marked their respective transitions in Fig. 2. Along with those, H_{α} and H_{β} atomic lines of hydrogen is also well apparent at wavelength 656.28 nm and 486.81 nm respectively.

i. Experimental estimation of temperature:

Figure 3 (a) represents the experimental calibrated data fitted of nitrogen first positive system corresponding



Fig. 2 Identified molecular bands of nitrogen along with H_{α} and H_{β} lines.



Fig. 3 Experimentally measured temperature with Boltzmann plots for $\Delta v = 4$ band. (a) Experimentally measured data and Gaussian fittings of each transition, (b) Boltzmann plot.

Table 1 Wavelength data for $\Delta v = 4$.

$\Delta v = 4$ <i>v'-v''</i>	Wavelength (nm)		$\frac{Transition \ Probability}{\times \ 10^4 \ (sec^{-1}) \ [14]}$
	[14]	Observed	
13-9	569.6	567.8	11.0
12-8	574.4	574.1	10.0
11-7	579.2	578.5	9.5
10-6	584.2	583.5	8.1
9-5	589.4	588.8	6.4
8-4	594.7	594.0	4.6
7-3	600.1	600.1	2.9
6-2	605.7	605.1	1.5
5-1	611.4	612.8	0.6

to $\Delta v = 4$, falling in the wavelength range of 560 - 620 nm. The nine vibrational transitions are tabulated in Table 1. The spectrum is fitted with multi-Gaussian, after subtracting the background. The resultant intensity peak values

Table 2 Wavelength data for $\Delta v = 3$.

$\Delta v = 3$ v'-v''	Wavelength (nm)		$\begin{array}{l} \mbox{Transition Probability} \\ \times \ 10^4 \ (sec^{-1}) \ [14] \end{array}$
	[14]	Observed	
10-7	630.9	628.2	7.6
9-6	638.0	637.9	9.1
8-5	645.4	644.7	9.9
7-4	653.0	652.7	9.6
6-3	660.8	660.1	8.2
5-2	668.9	668.3	5.9
4-1	677.2	676.4	3.5
3-0	685.8	685.0	1.1

are then used to construct the Boltzmann plot and the vibrational temperature is deduced. The vibrational upper levels energies are calculated using equation (2) by taking $w_e = 1733 \text{ cm}^{-1}$ and $w_e x_e = 14.412 \text{ cm}^{-1}$ for B³ \prod_{α} transition [13]. Figure 3 (b) shows the required Boltzmann plot for transition corresponding to $\Delta v = 4$ of the N₂ first positive system, where the symbols are the data points, and the solid line is the linear best fit to the data. The slope m = -3.09×10^{-4} , gives the vibrational temperature ~4680 K using equation (3). Similarly, the Nitrogen first positive system corresponding to $\Delta v = 3$, falling in the wavelength range of 620-680 nm is shown in Fig. 4(a). Total eight vibrational transitions are identified and fitted with multi-Gaussian. The data of wavelength and transition probability is tabulated in Table 2. With this, the Boltzmann plot has been constructed and it is shown in Fig. 4 (b). The slope $m = -1.82 \times 10^{-4}$, gives the vibrational temperature ~7931 K using equation (3). Again, the nitrogen first positive system corresponding to $\Delta v = 4$, falling in the wavelength range of 700 - 800 nm, is shown in Fig. 5 (a). Here, total nine vibrational transitions are identified and fitted with multi-Gaussian. The data for wavelengths are tabulated in Table 3. With this band information, the Boltzmann plot has been constructed as depicted in Fig. 5 (b). The slope $m = -1.84 \times 10^{-4}$, gives the vibrational temperature ~7851 K using equation (3). Here, it is seen that $\Delta v = 2$ and 3 gives temperature ~ 7800 K -8000 K, which is different from the temperature estimated from $\Delta v = 4$ band. Although the vibrational temperature

in the B-triplet state should be same as the emission shares the same upper level.

ii. Simulated estimation of temperature:

A MATLAB based code has been developed to simulate the band spectra of nitrogen 1PS. For each band, the code requires mainly the transition probability and wave-

Table 3 Wavelength data for $\Delta v = 2$.

$\Delta v = 2$ <i>v'-v''</i>		Wavelength (nm)	$\begin{array}{l} \text{Transition} \\ \text{probability} \\ \times \ 10^4 \ (\text{sec}^{\text{-1}}) \\ [14] \end{array}$
	[14]	Observed	
7-5	714.7	713.5	2.2
6-4	725.5	725.0	4.6
5-3	736.8	735.9	7.0
4-2	748.4	747.7	8.4
3-1	760.6	759.9	7.6
2-0	773.2	772.4	4.2



Fig. 4 Experimentally measured temperature with Boltzmann plots for $\Delta v = 3$ band (a) Experimentally measured data and Gaussian fittings of each transition, (b) Boltzmann plot.

length information as the background data. Through which it generates the maximum intensity value of each transition using equation (1). Here the temperature value in equation (1) is the variable parameter, which affects the line shape profile. After this, the intensity is then plotted with Gaussian line shape profile function as shown in Fig. 6. For $\Delta v = 4$ band, fixed FWHM ~1.7 nm is taken for each vibrational transition. The FWHM is taken as fixed parameter for each vibrational transition in a single band as it was seen in the previous studies that the line shape profile of N_2 molecular band is less sensitive to rotational temperature, i.e., the rotational FWHM value [15]. Also, it is seen here that there is not large difference in experimentally measured band's FWHM values in a single band. The simulated spectra were iterated for different temperatures to match the experimental one and the vibrational temperature is estimated to be 4000 K as shown in Fig. 6 (a). It is seen that the spectra at higher wavelength side of the



Fig. 5 xperimentally measured temperature with Boltzmann plots for $\Delta v = 2$ band (a) Experimentally measured data and Gaussian fittings of each transition, (b) Boltzmann plot.



Fig. 6 Simulated spectra for band (a) $\Delta v = 4$, (b) $\Delta v = 3$ and (c) $\Delta v = 2$.

band is not matching well. For $\Delta v = 3$ band with fixed FWHM of 2.6 nm for each transition, again the temperature is iterated and it is found ~7900 K at best-matched condition with experimental data as shown in Fig. 6(b). Again, there is mismatch in the experimental and simulated spectra at the lower wavelength side of the band. Similarly, for $\Delta v = 2$ band the FWHM is kept at 3.1 nm fixed for each transition, the temperature is again iterated. At temperature of \sim 7600 K as shown in Fig. 6 (c). The experimental and simulated results of this band is matched very well. Also, the temperature measured here is close to the value obtained by the Boltzmann plot method which was ~7851 K for the same band. As shown in Fig. 6 (c), for $\Delta v = 2$, band is having best-matched between the experimental and simulated results. However, there is some discrepancy in the measurements of vibrational temperature using the other two bands viz. $\Delta v = 4$ and 3, as shown in Figs. 6 (a) and 6 (b), respectively. This ambiguity is discussed in the next section.

5. Discussion

It is observed in the results (section 4) that the nitrogen molecular bands corresponding to B-triplet state present between 560 - 700 nm which shows mismatch in the simulated band fittings (Figs. 6 (a) and 6 (b)). In addition to this, there is a discrepancy in experimentally estimated temperature for $\Delta v = 4$ band using both Boltzmann plot method and also simulated estimation as compared to the other two bands viz. $\Delta v = 2$ and 3. This leads to the hypothesis that may have been perturbed by other spectral lines. To see the effect of band perturbed by other spectral lines, the pure hydrogen plasma was produced with the same experimental conditions. The experimental spectra for the same were recorded along with the Fulcher- α band having transition $d^3 \prod_{u} \rightarrow a^3 \sum_{g}^{+}$ between 560 - 700 nm of wavelength range [16]. The Q-branch lines of Fulcher- α are recognized and are marked in Fig. 7, as Q, 601.92 nm (0,0), 613.16 nm (1,1), 623.07 nm (2,2), and 634.64 nm (3,3). As a result, it becomes clear that these bands are mainly affecting the nitrogen molecular band measurements between 600 to 650 nm of wavelength range. However, this affects more to the band present at 600 nm. Hence, there is a disagreement in the temperature of the band falling at this wavelength, 560-620 nm ($\Delta v = 4$) ($T_{vib} \sim 4600$ K as shown in Fig. 3 and 5(a)). The effect of this can be minimized by subtracting the Fulcher band from the main measured nitrogen band spectra. The result of this is now reflected in the experimentally estimated temperature, which is now ~8141 K as shown in Fig. 8 and is close to the measurement in which the band $\Delta v = 2$, is not influenced by any other lines. Due to this correction, the estimated temperatures, using all the three bands become almost equal within the experimental errors. Hence the discrepancy is removed in the experimentally measured temperatures. However, it is to be noted that the contribution from the



Fig. 7 Identified molecular bands of hydrogen.



Fig. 8 Estimated temperature using Boltzmann plot method for $\Delta v = 4$ band after suppressing effect of Fulcher band in measurement.



Fig. 9 Simulated spectra for band (a) $\Delta v = 4$ and (b) $\Delta v = 3$.

molecular hydrogen bands may be modified if the rovibrational temperature of the hydrogen changes between with and without the existence of nitrogen. The correction of N_2 spectra by using Fulcher- α , has been applied in the simulated estimation technique. For this purpose, the lines present in Fulcher bands are incorporated in the MATLAB codes. The simulated bands were now better matched with experimental data at temperature $T_{vib} = 7600$ and 7900, for $\Delta v = 4$ and 3, respectively, as shown in Figs. 9 (a) and 9 (b).

6. Summary

In this paper, the measurement of the nitrogen molecular bands is presented. The experiments are carried out in the PACVD chamber and visible spectrum are recorded using a spectroscopic system in the wavelength range of 350-900 nm. It is observed that the nitrogen first positive $(B^3 \prod_g -A^3 \sum_u^+)$ system is dominated in this RF generated nitrogen plasma. The dominated identified bands are $\Delta v = 4$, 3 and 2, between 560 to 800 nm of wavelength range. Additionally, there is presence of atomic lines of hydrogen and second positive system of nitrogen at lower wavelength side. To estimate the gas temperature, it was measured for all the three bands. Here, two techniques was adopted for this purpose, one was based on the Boltzmann plot method and another was the simulation of molecular band spectra. During measurements the discrepancy in temperature was observed, especially between $\Delta v = 4$ and $\Delta v = 2$ to 3, when one uses the Boltzmann plot method. Moreover, there was an issue of matching between the experimental and simulated data for $\Delta v = 4$ and 3. It is found that these spectrums are affected by Fulcher- band. This band is then subtracted from the measured nitrogen band to estimate the temperature. With this technique, the estimated vibrational gas temperature using both the methods for different nitrogen bands are \sim 7000 - 8000 K and the ambiguity is resolved.

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