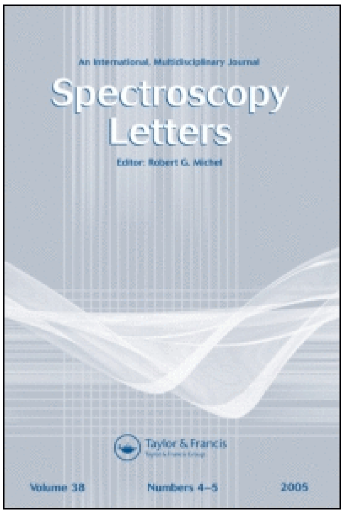

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Vibrational Distribution of $N_2(C, \nu)$ State in a Pulsed-DC Generated N_2–Ar Glow Discharge

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ABSTRACT The dependence of vibrational distributions (VDs) of $N_2(C, \nu)$ state on argon fraction and filling pressure was investigated in terms of integrated band intensities of second positive system $(C, \nu' \rightarrow B, \nu'')$ of N_2. The vibrational temperature was determined from slope of the Boltzmann fit of these VDs and found to be strongly dependent on argon fraction in the discharge. The spectroscopic results suggested that collisional transfer of energy from Ar $(^3P_{2,0})$ metastable atoms to $N_2(X, \nu)$ ground state is the main excitation pathway for population of $N_2(C, \nu)$ state.

KEYWORDS argon metastable atoms, optical spectrum, Penning effect, vibrational distribution

INTRODUCTION

The vibrational distribution provides information about the relative rates of vibration-vibration and vibration-translation energy exchange processes in the discharges.[1] Mostly, in nitrogen discharges, it is found that there is a strong correlation between the vibrational distribution function (VDF) of molecules in the electronically excited states and the electron density, while the ratio of populations of the excited states $N_2(B\ ^3\Pi_g, \nu)$ and $N_2\ (C\ ^3\Pi_u, \nu')$ is related to the electron temperature.[2] Moreover, nitrogen plasmas have wide range of application in nitriding of various materials, especially Fe-based alloys to improve their surface properties including surface hardness, wear, corrosion resistance, and fatigue strength.[3,4] The large number of discharge parameters that can arbitrarily be selected, makes it attractive for the growth of specific structures and properties, unlike conventional nitriding.[5] The process efficiency of the discharge depends on concentration of active species and the electron density, which in turn are strongly coupled with the vibrational distribution of the electronically excited states. Therefore, there is great interest in the study of nitrogen discharges containing inert gases that serve as a catalyst for active species generation.[6] The metastable states of atoms and molecules, owing to their ability to accumulate a great amount of energy, play a significant role in the production of active species that can be effective in various chemical and physical

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processes.[7] Therefore, inert gases such as argon, neon and helium, which have a plenty of metastable states in nitrogen plasma affect the vibrational distribution and consequently the concentration of active species through the Penning reaction.[8] The vibrational distribution of electronically excited states is of fundamental importance in plasma processes since the vibrational excitation due to its adiabatic character can trap energy and thus plays the role of energy reservoir, which is important for chemical reactions in plasma.[9] The inert gases play an important part in the kinetics of the nitrogen plasma and provide a better control on the vibrational excitation of the electronic states.[10] The $N_2(C\,^3\Pi_u,\nu)$ and $N_2^+(B\,^2\Sigma_u^+,\nu)$ are radiative states emitting, respectively, the first negative band system and the second positive band systems. Therefore, dependence of vibrational population of $N_2(C\,^3\Pi_u,\nu)$ state on argon percentage in nitrogen discharge will help to explore the role of argon metastable atoms in particle collision processes, plasma kinetics and production of active species.

Optical emission spectroscopy provides a non-intrusive means of studying reactive plasmas.[11,12] The basic premise of this technique is that the emission intensity of the excited states of plasma species is proportional to their population densities provided that the population dynamics are governed by the direct electron impact excitation from the ground state without any multistage excitation, ionization or self-absorption.[13–15] Thus plasma spectroscopy is an indispensable diagnostic technique in plasma processing and technology as well as in fundamental research methodology.

This work is the extension of our previous work,[16] in which plasma electron temperature and its resulting effect on the generation of active species (excited molecules and radicals) was studied as a function of discharge parameters such as input power, filling pressure, and plasma gas composition. The results showed that electron temperature can be raised significantly by mixing argon in nitrogen plasma, which in turn enhances the concentration of active species through collisional transfer of energy. Moreover, in N_2–Ar gas mixture plasma; there was a high electronic excitation of N_2 from energy transfer of argon metastable atoms. The experimental findings were interesting and motivating for selective vibrational excitation of $(C\,^3\Pi_u,\nu)$ state through collisions with metastable atoms and molecules in the N_2–Ar discharge. The emission intensity of second positive band system $(C,\nu'\rightarrow B,\nu'')$ of N_2 was obtained from the optical spectrum recorded at different gas compositions under different discharge conditions and then correlated to population of $(C^3\Pi_u,\nu)$ state.

EXPERIMENT

A 50 Hz pulsating-dc electrical power was provided to excite and sustain discharge in a parallel plate configuration of electrodes housed in a cylindrical stainless steel vacuum chamber of 40 cm diameter and height. The diameter of each electrode was 7.5 cm and a spacing of 6 cm. The side and back of the electrodes were covered with ceramic casing to prevent the discharge with the wall of the chamber. The discharge was sustained in an abnormal glow mode, usually used for materials processing.[15] The power was coupled to the discharge through the inductive load, which serves as a current limiter during the discharge. Prior to admitting the nitrogen and argon gases, the chamber was evacuated down to 10^{-5} mbar using a rotary vane pump and oil diffusion pump. The flow of nitrogen and argon gases was monitored with mass flow meters whereas pressure in the chamber was recorded by using capsule type dial gauge. Optical emission spectroscopy was carried out using a computer-controlled detection and data acquisition systems comprising McPherson-2061, one meter monochromator having a diffraction grating with 1200 grooves/mm and spectral resolution of 0.01 nm coupled with a side window photo-multiplier tube (PMT-9781B), and an auto ranging pico-ammeter (Keithley-485). The width of the entrance slit, focal length and aperture of the monochromator were 5 μm, 1 m and $f/7$, respectively. The entrance slit of the monochromator was positioned at a distance of 15 cm from the optical window of fused silica to collect a sufficient light without light-collecting optics. More details of plasma generation source along with optical detection and data acquisition systems are presented elsewhere.[16] The wavelength calibration of the monochromator was performed by using a mercury lamp and corrected for the spectral response of the grating and photomultiplier tube. Argon gas was admixed with nitrogen in a controlled manner and

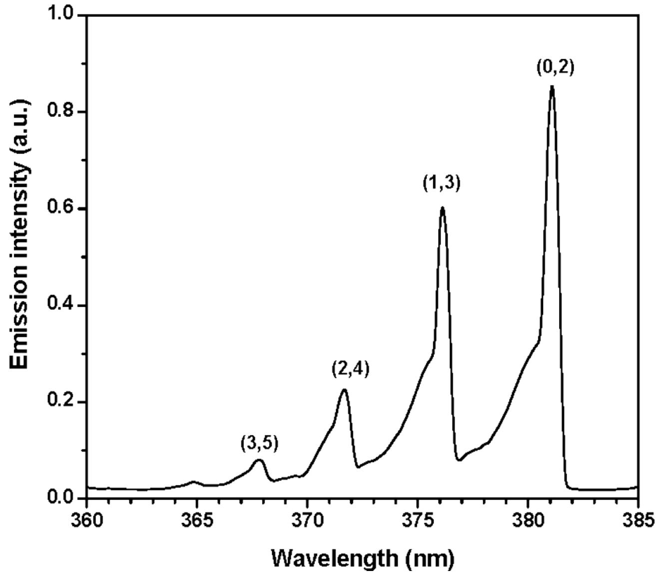


FIGURE 1 **Emission spectrum from N_2-20% Ar discharge at a filling pressure of 5mbar, input power of 300 watts and total flow rate of 100 sccm. The sequence of four peaks with the vibrational quantum number difference $\Delta\nu \equiv \nu' - \nu'' = -2$ (0–2 at 380.5 nm, 1–3 at 375.5 nm, 2–4 at 371.1 nm and 3–5 at 367.2 nm labeled in spectrum) was chosen to determine the T_ν, of $N_2(C, \nu')$] state.**

emission spectra (300 to 800 nm) were recorded as a function of argon fraction (20% to 80%) and filling pressures (5 to 9 mbar), keeping the corresponding discharge parameters constant. A typical emission spectrum of N_2-20% Ar discharge at a filling pressure of 5 mbar, input power of 300 watts and total flow rate of 100 sccm is shown Fig. 1. The sequence of four peaks with the vibrational quantum number difference $\Delta\nu \equiv \nu' - \nu'' = -2$ (0–2 at 380.5 nm, 1–3 at 375.5 nm, 2–4 at 371.1 nm and 3–5 at 367.2 nm labeled in spectrum) was chosen to determine the vibrational distribution of $N_2(C,\nu')$ state and accordingly the vibrational temperature T_ν, from the Boltzmann fit.

POPULATION OF $N_2(C'\ \nu)$ STATE

The $N_2(C^3\Pi_u,\nu')$ state can be populated by electron impact excitation from the ground state $N_2(X\,^1\Sigma_g^+,\nu)$ of the neutral N_2 molecule and first metastable state $N_2(A\,^3\Sigma_u^+\nu'')$ produced by electron impact excitation[4,17] via

$$N_2(X,\nu) + e \longrightarrow N_2(C,\nu') + e$$
$$N_2(X,\nu) + e \longrightarrow N_2(A,\nu'') + e$$
$$N_2(A,\nu'') + e \longrightarrow N_2(C,\nu') + e$$

The other mechanism of population of $N_2(C,\nu')$ state are associative excitation and energy pooling reactions.[17]

$$N_2(X,\nu) + N_2(A,\nu'') \longrightarrow N_2(C,\nu') + N_2(X,\omega)$$
$$N_2(A,\nu) + N_2(A,\omega) \longrightarrow N_2(C,\nu') + N_2(X,\nu)$$

Mixing of argon in nitrogen plasma may also populate the N_2 (C, ν') excited state by the transfer of internal energy from a metastable state of argon atoms to the ground state of the nitrogen molecules. Moreover, the argon metastable states ($^3P_{2,0}$) are at the higher energies 11.55 and 11.72 eV, respectively, than the threshold excitation energy (11.1 eV) of N_2 (C,ν') state. Therefore, on adding argon in nitrogen plasma, a significant enhancement in the population of $N_2(C,\nu')$ state, and consequently the emission intensity of the second positive band system of N_2 is expected by effective excitation of N_2 through inelastic collisions of argon metastable atoms.[16]

$$N_2(X,\nu) + Ar_m^*(^3P_{2,0}) \longrightarrow$$
$$N_2(C,\nu') + Ar(\varepsilon > 11.1\,\text{eV from}\,N_2(X,\nu=0))$$

The subsequent radiative decays of $N_2(C,\ \nu')$ excited state emit characteristic band of second positive system (SPS).

$$N_2(C,\nu') \longrightarrow N_2(B,\nu'') + h\nu$$

The vibrational level population in the N_2 (C,ν') state can be derived from emission intensities of vibrational bands of the SPS of a nitrogen emission spectrum. The emission intensity of the vibrational band corresponding to a transition (C, $\nu' \to$ B,ν'') is given by[11]

$$I(C,\nu' - B,\nu'') = c(\lambda_{\nu'-\nu''})[N_2(C,\nu')]$$
$$+ A(C,\nu' \longrightarrow B,\nu'')/\lambda_{\nu'-\nu''}$$

where I (C,ν'-B,ν'') is an experimentally measured emission intensity of the transition, $c(\lambda\nu' - \nu'')$ is the spectral response of the optical detection system at wavelength, $\lambda_{\nu'-\nu''}$, [$N_2(C,\nu')$] is the density of nitrogen molecules at level ν' and A(C,$\nu' \to$B, ν'') is the radiative transition probability, which can be found in.[19] Therefore, the vibrational distribution of $N_2(C,\ \nu')$ radiative state can be determined readily from the emission intensity of the respective transition together with corresponding bandhead wavelength and transition probability. From the measured vibrational distribution (VD), it is possible to deduce a vibrational temperature (T_{ν}). As a matter of fact the semi-log plots (Fig. 2) show that the VDs are close to a Boltzmann distribution, up to $\nu' = 0$–3. However, for higher vibrational levels the distribution of vibrational population has a more complicated shape.[20] Since in low temperature plasmas only a few first vibrational levels are significantly populated, the Boltzmann plot method is therefore applicable to determine the vibrational temperature. Accordingly, the vibrational temperature T_{ν} of $N_2(C, \nu')$ state can be determined from the slope of semi-log Boltzmann plot of relative population of N_2 (C, ν') state.[9] The semi-log Boltzmann plots for several N_2–Ar gas mixtures is presented in Fig. 3

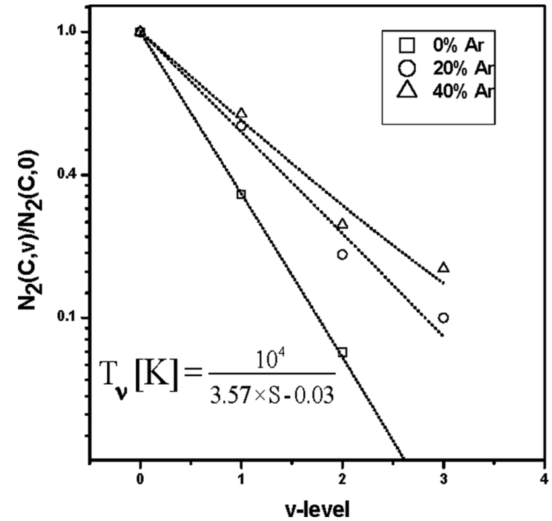
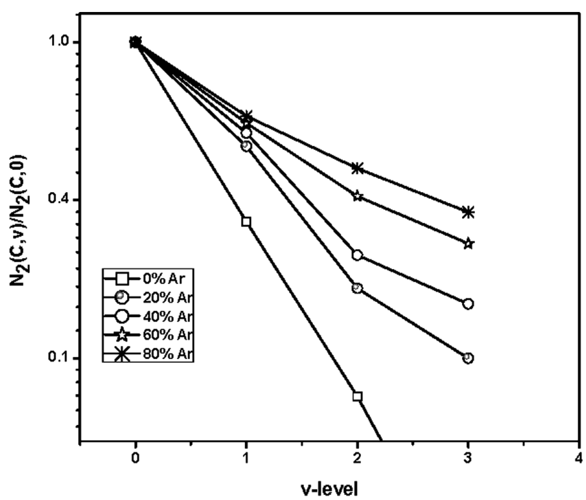
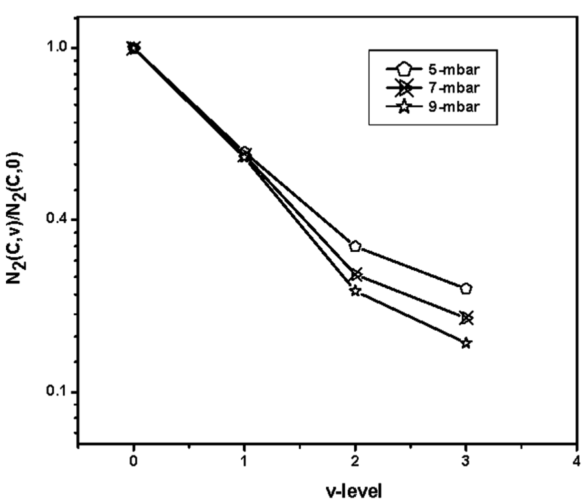


FIGURE 3 The semi-log Boltzmann plots for $N_2(C, \nu')$ relative vibrational distribution along with the relation between T_{ν} and absolute value of Boltzmann slope S in log-scale.[9]

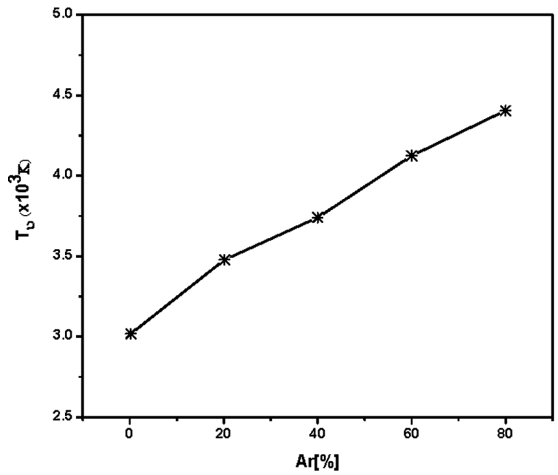


(a)

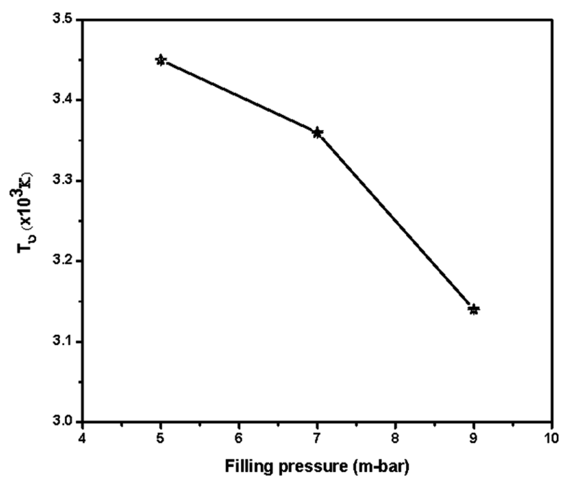


(b)

FIGURE 2 $N_2(C, \nu')$ relative vibrational distribution (a) for various Ar fractions in the mixture at a filling pressure of 5-mbar and input power of 300 watts, and (b) for various filling pressure at an argon fraction of 20% and input power of 300 watts. The relative population was deduced from the integrated band intensities of second positive system $(C, \nu' \rightarrow B, \nu'')$ of N_2.



(a)



(b)

FIGURE 4 Vibrational temperature T_{ν} (a) as a function of argon fraction in the mixture at a filling pressure of 5-mbar and input power of 300 watts, and (b) as a function of filling pressure at an argon fraction of 20% and input power of 300 watts.

whereas T_ν derived from their respective slopes is presented in Fig. 4.

RESULTS AND DISCUSSION

It is well-known that argon atom has $^3P_2$ and $^3P_0$ triplet metastable states with energies 11.55 eV and 11.72 eV, respectively, and its ionization energy is 15.7 eV. Nitrogen molecule, on the other hand has one triplet metastable state $(A\,^3\Sigma_u^+)$ at 6.20 eV and some singlet metastable states with energies ranging upto 9.30 eV. Its ionization energy is 15.57 eV. The metastable atoms and molecules may therefore be expected to play an important part in the energy exchange process occurring in the discharge. Many of the spectral phenomena resulting from influence of inert gases have been successfully explained with the help of inelastic collision processes, involving the metastable atoms and molecules. Nevertheless, there is still a room for further investigations on the role of metastables in the excitation process of molecular gases. In N_2–Ar gas mixtures, metastable argon atoms may act as energy carriers to cause efficient excitation of the nitrogen molecule from the ground state $N_2(X, \nu)$ to excited state $N_2(C, \nu')$ through inelastic collisions. Fig. 2 shows that for pure N_2 discharge the measured $N_2(C, \nu')$ distribution, which is derived from the emission intensities of vibrational bands of second positive system of N_2 can be characterized by Boltzmann distribution. However, by adding argon into nitrogen a remarkably different populations of vibrational levels of $N_2(C, \nu')$ is reached, and higher vibrational levels of $N_2(C, \nu')$ state are predominantly overpopulated. Since the excitation of higher vibrational levels by electron impact is negligible in such low temperature plasmas, the considerable population of these levels in N_2–Ar mixture plasma can be attributed mainly to a high excitation transfer of N_2 from metastable argon atoms. Moreover, the threshold excitation energy of $N_2(C, \nu')$ radiative state is 11.1 eV, which is slightly lower than those of the argon metastables $^3P_2$ (11.55 eV) and $^3P_0$ (11.72 eV). Consequently, the $N_2(C, \nu')$ radiative state can be populated efficiently by transfer of energy from metastable argon atoms to $N_2(X, \nu)$ ground state through inelastic collisions. This additional excitation channel leads to a non-linear population of $N_2(C, \nu')$ state and consequently the emission intensities of the vibrational bands of SPS of N_2. Furthermore, energy of the argon metastables (11.55 eV and 11.72 eV) is lower than the first ionization energy of the N_2 molecule (15.57 eV), so ionization mechanism of the N_2 is not expected to be influenced considerably by argon metastables. However one ought to take into account the contribution resulted from indirect excitation pathway of $N_2(C, \nu')$ state through electron impact excitation of $(A\,^3\Sigma_u^+)$ metastable state, which is also influenced by changing electron temperature with argon fraction and filling pressure. Since the increase in argon fraction in the discharge can lead to an increase in electron temperature owing to its higher ionization potential.[16] This change in the plasma can not only cause an increase in argon metastable density but can also lead to an increase in the rate of both reactions involved in the indirect excitation pathway thereby increasing the $N_2(C, \nu')$ density. Similarly, an increase in pressure will lead to a decrease in electron temperature,[16] and subsequently a decrease in the argon metastable density in the discharge thus reducing the $N_2(C, \nu')$ density. This latter effect can also be attributed to the decrease in the rate constant of both reactions for the indirect excitation pathway. Thus it is evident that the enhancement in the preferential excitation of $N_2(C, \nu')$ state may come about through any of the dominant excitation pathways that is indirect excitation or Penning excitation, acting either singly or jointly. Interactions between metastable argon atoms and N_2 molecules are also possible resulting in the Penning dissociation, but such reactions are not expected to play an important role in low temperature plasmas due to their energetic nature. The decrease in relative population of $N_2(C, \nu')$ state with increasing pressure may also be explained by the fact that excitation efficiency of the discharge is reduced owing to increase in collisional losses of electron kinetic energy. As a result, the number of electrons, which are available to promote the argon atoms to their metastable states, is reduced that in turn contribute to the preferential excitation of $N_2(C, \nu')$ state through inelastic collisions together with indirect excitation of $N_2(C, \nu')$ state involving $(A\,^3\Sigma_u^+)$ metastable state.

Fig. 3 depicts the semi-log Boltzmann plots for relative vibrational distribution of $N_2(C, \nu')$ state together with the relation between vibrational temperature T_ν and absolute value of Boltzmann slope

S in log-scale,[9] used to calculate the T_ν for different argon percentages in the gas mixture plasma.

Fig. 4 shows that the vibrational temperature T_ν of the N_2(C, ν') state increases with argon fraction and decreases with filling pressure. As stated earlier, the electron temperature and consequently the argon metastable density increases by increasing the argon fraction in the mixture. As a result preferential excitation of N_2(C, ν') state is come about by both direct excitation pathway involving metastable argon atoms and indirect excitation pathway involving $(A\,^3\Sigma_u^+)$ metastable state of N_2 and consequently the vibrational temperature T_ν. The decrease in vibrational temperature with filling pressure can be attributed to lesser number of energetic electrons able to promote the argon atoms to metastables, which in turn participate in energy transfer process along with indirect excitation of N_2(C, ν') state via electron impact excitation of $(A\,^3\Sigma_u^+)$ metastable state of N_2.

Effect of helium gas mixing on the electron temperature and selective excitation of N_2(C, ν') and $N_2^+(B, \nu')$ states has been already studied and reported.[21] The experimental results showed the same trend regarding the enhancement of electron temperature and preferential excitation of N_2(C, ν') state as observed in the case of argon. However, effect on electron temperature and excitation of $N_2^+(B, \nu')$ states was pronounced in the case of helium when compared with argon, which may be attributed to higher metastable energies of helium. Furthermore, it is found that electron temperature and consequently population of radiative states can be controlled effectively by using both the inert gases in the nitrogen discharge.

CONCLUDING REMARKS

Spectroscopic results on the vibrational distribution of second positive system (C, $\nu' \rightarrow$ B, ν'') of N_2 is presented to characterize the excitation processes of N_2(C, ν') as a function of argon fraction in the N_2 discharge and filling pressure. The main thrust of the work is to analyze the role of argon metastable atoms on the preferential excitation of N_2(C, ν'). There is a strong and peculiar dependence of the vibrational distributions of N_2(C, ν') state on argon metastables in the discharge. On mixing of argon a significant enhancement in the population of higher vibrational levels of N_2(C, ν') state is found, which may be attributed to both direct excitation pathway involving metastable argon atoms and indirect excitation pathway involving $(A\,^3\Sigma_u^+)$ metastable state of N_2. Consequently, the vibrational temperature T_ν increases significantly with increasing argon fraction in the discharge while slight decrease in vibrational temperature is observed as a result of increasing filling pressure.

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