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MEASUREMENT OF ROTATIONAL TEMPERATURES IN HIGH-PRESSURE MICROHOLLOW CATHODE (MHC) AND CAPILLARY PLASMA ELECTRODE (CPE) DISCHARGES [1]

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We report the results of rotational temperature (T_R) measurements in high-pressure (400 Torr) microhollow cathode (MHC) discharges in Ne with a trace admixture of N_2 using the unresolved N_2 second positive band and the N_2^+ first negative band. Significantly different values for T_R were obtained for respectively N_2 and N_2^+. The rotational temperatures obtained from the analysis of the N_2 band system are slightly above room temperature, increase with increasing discharge current, and may be interpreted as the gas kinetic temperature in the MHC discharge plasma. By contrast, the analysis of the N_2^+ band system yielded rotational temperatures of more than 900 K, which are the result of collisional reaction processes leading to the formation of rotationally excited N_2^+ ions. The effective lifetime of these ions is comparable to the rotational relaxation time, so that the ions retain much of their rotational excitation prior to emission and are not in thermal equilibrium with the bulk gas. Thus, the T_R values obtained for N_2^+ cannot be equated with the gas kinetic temperature in the plasma. We also carried out a rotational analysis of the unresolved N_2 second positive system emitted by an atmospheric-pressure capillary plasma electrode (CPE) discharge in ambient air and measured a temperature of 545 K. This temperature may be close to the gas kinetic temperature as the rotational analysis utilized N_2 emissions from inside the capillary which is the region of highest plasma density and highest gas temperature in a CPE discharge.

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1 Introduction

Emission spectroscopy is a powerful tool to obtain information about the important parameters that characterize non-equilibrium discharge plasmas at both low and high pressure (for a detailed

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review of this field and the literature up to the mid 1990s, see Griem [1]). Emissions of diatomic species such as N_2, N_2^+, OH, and NO can be used to determine translational, vibrational, and rotational temperatures of the plasma species. Emission spectroscopy in conjunction with electron-impact cross sections can also be used to obtain at least qualitative information about the energy distribution of the plasma electrons. Electron densities can be determined from the Stark broadening of the H Balmer-β line or from the NO δ-band emission spectrum. The rotational temperature T_R is perhaps the most frequently determined quantity from plasma optical emission spectroscopy (OES) as its determination may reflect the gas temperature in the plasma and/or provide insight into the reaction kinetics of important plasma species [2]. The second positive band system of N_2 ($C\ ^3\Pi_u \rightarrow B\ ^3\Pi_u$) and the first negative band system of N_2^+ ($B\ ^2\Sigma_u^+ \rightarrow X\ ^2\Sigma_g$) are perhaps the most frequently analyzed molecular emission systems in OES studies. The reason is that these emissions are readily observed in many discharge types as long as there is a trace of N_2 in the discharge gas mixture and that the molecular constants describing these two transitions are well-known [3], so that synthetic model spectra of these two band systems can be calculated easily and with high accuracy. Assuming that the emitting N_2 molecules or N_2^+ ions can be described by a Maxwell-Boltzmann distribution characterized by a single rotational temperature T_R, this temperature can be determined from a fit of the measured emission spectrum, usually from a single, isolated vibrational band, to a synthetic spectrum with T_R as the only free parameter. This can be accomplished, for instance, by recording the fully rotationally resolved structure of the band (i.e. by recording the emission intensity of the individual rotational lines of the band) and by fitting the intensities of the rotational lines to a "rotational Boltzmann factor" from which one can obtain T_R. As an alternative to recording fully rotationally resolved spectra, one can record the unresolved envelope of vibrational band with high statistical accuracy and then obtain T_R from a "best-fit" of the envelope of the measured spectrum to a calculated band envelope, again with the rotational temperature as the only free parameter. However, the calculation of the band envelope requires the convolution of the calculated intensity of each rotational lines with a monochromator slit function, which must determined with high precision in a separate experiment using a narrow atomic line emission.

The rotational temperatures determined in the kind of OES studies described above reflect the rotational population of the emitting species. If the emitting species are in equilibrium with the bulk gas in the plasma, then this temperature can be interpreted as the gas kinetic temperature in the plasma. Trace amounts of N_2 are frequently added to plasma gas mixtures that do not contain N_2 in an effort to determine the gas kinetic temperature from a rotational analysis of the N_2 or N_2^+ emissions. However, great care must be exercised in the interpretation of such measurements. The measured rotational N_2 or N_2^+ temperature can only be equated with the gas kinetic temperature in the plasma, if the emitting species are in equilibrium with the bulk gas. Situations may also arise (see e.g. Wiesemann and co-workers [2] and references therein to earlier work) where two rotational temperatures are needed to fit a measured emission band, usually a low T_{R1} representing rotationally "cold" species and a higher T_{R2} representing rotationally "hot" species. The presence of rotationally "hot" species under those circumstances is usually the result of more or less complex collisional interactions involving the various plasma constitutents that leave the emitting species in rotationally excited states.

In the present study we determined rotational temperatures for N_2 and N_2^+ in high-pressure (400 Torr) microhollow cathode (MHC) discharges in mixtures in Ne with a trace admixture of N_2 employing a rotational analysis of the unresolved N_2 second positive band and the N_2^+ first negative band. Significantly different values for T_R were obtained for respectively N_2 and N_2^+. The relation of these rotational temperatures to the gas kinetic temperature and to the reaction kinetics leading to the formation of the emitting N_2 and N_2^+ species will be discussed. We also carried out a rotational analysis of the unresolved N_2 second positive system emitted by an atmospheric-pressure capillary plasma electrode (CPE) discharge in ambient air.

2 Experimental Details

The details of the two high-pressure discharge plasmas used in the present studies have been described in earlier publications [4-7] and only a brief summary will be given here.

2.1 Microhollow Cathode (MHC) and Capillary Plasma Electrode (CPE) Discharges

A hollow cathode (HC) discharge device consists of a metallic cathode with a hole in the center and an arbitrarily shaped metallic anode. The two electrodes are separated by an insulator with a hole in it that is similar in size and shape to the hole in the cathode. When a discharge is ignited in such a configuration, the negative glow is spatially confined in the cathode cavity. The electric field in this region creates a trough which is responsible for the strong acceleration of the electrons and may cause an oscillatory motion of the electron in the trough ("pendulum electrons" [8-11]. These "pendulum electrons" can undergo many ionizing collisions with the background gas thus creating a high-density plasma which emits intense radiation. The hole diameter D of a HC discharge device is inversely proportional to the operating pressure up to about 10 Torr/D (in cm) for noble gases and N_2 [12, 13], so that atmospheric-pressure operation requires a hole diameter of the order of 100 μm (micro hollow cathode or MHC discharge).

The MHC discharge device used in the present study has been described in detail in previous publications [4, 5] and only a brief description will be given here. The electrodes of our MHC discharge device are made of 0.1 mm thick molybdenum foils separated by a 0.25 mm spacer of an insulator with a hole of about 150 μm diameter in the cathode, the dielectric, and in the anode. Supply voltages were typically between 400 V to 700 V and sustaining (discharge) voltages were in the range of 200 V - 300 V depending on the gas, the pressure, and the actual geometry of the MHC discharge. Discharge currents I_{DIS} varied between 0.5 – 3 mA. The operating gas was high-pressure Ne (around 400 Torr) with a small (typically 1 %) admixture of N_2.

The basis for the atmospheric-pressure operation of the capillary plasma electrode (CPE) discharge is a novel electrode design that uses dielectric capillaries that cover one or both electrodes of a discharge device [6, 7], which in many other aspects looks similar to a conventional dielectric barrier discharge (DBD). The capillaries, with diameters in the range from 0.01 to 1 mm and length-to-diameter ratios of the order of 10:1, serve as plasma sources which produce jets of high-intensity plasma at atmospheric pressure under the right operating conditions. The plasma jets emerge from the end of the capillary and form a "plasma electrode" for the main discharge plasma. Under the right combination of capillary geometry, dielectric material, and exciting electric field, a steady state can be achieved. The placement of the tubular dielectric capillaries in front of the electrode(s) creates a discharge with properties distinctly different from those of a DBD. The CPE discharge displays two modes of operation when excited by pulsed dc or ac. When the frequency of the applied voltage pulse is increased above a few kHz, one observes first a diffuse mode similar to the diffuse glow described of a DBD as described by Okazaki and co-workers [14]. When the frequency reaches a critical value (which depends strongly on the L/D value and the feed gas), the capillaries "turn on" and a bright, intense plasma jet emerges from the capillaries. While a full understanding of the fundamental processes in the CPE discharge on a microscopic scale has not been achieved, it appears that the capillaries act as individual high-density plasma sources. The initial step is the formation of a streamer-like discharge inside each capillary, whose properties are critically determined by their interaction with the dielectric walls of the capillaries. The transition into an arc is prevented by the fact that the current through the capillaries is self-limiting, i.e. the gas density inside the capillary decreases with time due to gas heating which puts an upper limit to the conductivity as a result of gas starvation.

The CPE discharge device used in the present study consisted of a square 5 x 5 area of 25 capillary electrodes. The discharge was operated in ambient air and excited by AC voltage, 6.68 kV peak-to-peak, at about 20 kHz. The discharge current was about 5.3 mA per capillary and was kept constant for all studies involving the CPE discharge device.

2.2 Spectroscopic Measurements

Two different experimental set-ups were used for the spectroscopic measurements which will be described briefly in the following section.

The experimental set-up for the emission spectroscopic studies of the MHC discharge consists of a Bausch & Lomb double-grating monochromator equipped with three sets of fixed entrance and exits slits corresponding to full width at half maximum (FWHM) resolutions of 2 nm, 0.5 nm, and 0.2 nm. All measurements reported here were carried out with a 0.2 nm (FWHM) resolution. The MHC discharge was mounted directly to the entrance slit assembly of the monochromator. A wide-range cooled photomultiplier tube (PMT) was used to detect the N_2 and N_2^+ photons at the exit slit of the monochromator. The observed spectra were obtained by scanning the monochromator across the respective wavelength regions of the (1,2) band of the N_2 second positive system (around 354 nm) and the (0,0) band of the N_2^+ first negative system (around 391.5 nm), which were selected as they are free from contamination from adjacent emission features and recording the emission intensity as a function of wavelength under computer control. Great care was exercised to limit the photon count rate to the regime where the output signal of the PMT varied linearly with the intensity of the input signal in order to avoid saturation effects.

The experimental set-up for the emission spectroscopic studies of the CPE discharge was comprised of a SPEX high-resolution, scanning monochromator with variable entrance and exits slits in conjunction with a 1024 x 256 pixel CCD detector (Roper Scientific I-MAX - 1024). In the present work, the slit-width of both slits was set to 100 μm which corresponds to 0.25 nm resolution (FWHM). The useful spectral range of the spectrometer – CCD camera detection system spans the interval from 200 nm to 800 nm. To optimize the collection of light a lens (focal length f = 7.5 cm, diameter d = 4.5 cm) was used to focus the light from the capillary discharge array onto the entrance slit of the monochromator. The plasma reactor was positioned in such a way that the light was collected from the axis of a single capillary. Thus, we determined the rotational temperature from gas in the plasma region of highest plasma and current density. Emission spectra were recorded in the wavelength range from 210 nm to 350 nm covering the emissions of the NO γ-bands and the N_2second positive system. Only the results obtained from

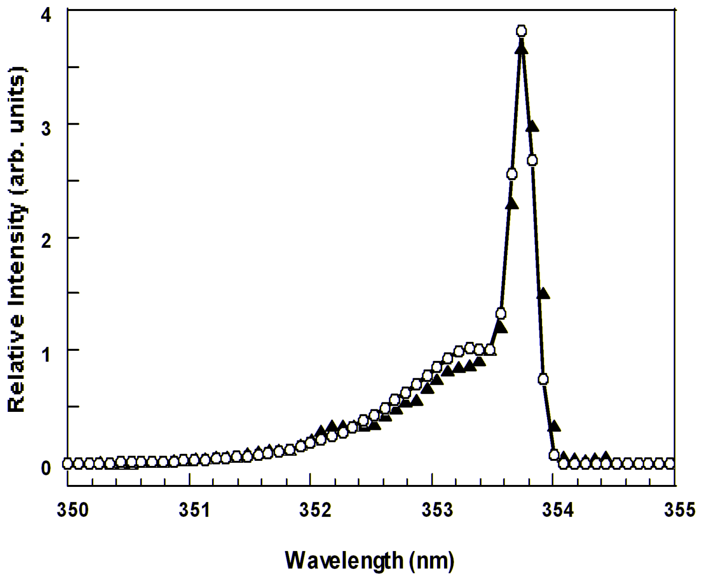

Fig. 1. Comparison of a measured spectrum of the (1,2) band of the N_2 second positive band recorded at a discharge current of 1 mA in a MHC discharge in 400 Torr Ne with a 1% admixture of N_2 (filled triangles) with a model spectrum corresponding to a rotational temperature of 375 K (open circles connected by a line).

the N_2 second positive system are reported here. The output of the CCD camera was routed into a PC for data storage and further analysis. As before, the signal intensity was chosen to be sufficiently low to avoid saturation effects of the CCD detector.

3 Results and Discussion

The calculation of the line intensities for the synthetic spectra of both the N_2 second positive system and the N_2^+ first negative system is straightforward [15] using the molecular constants as reported by Laux and Kruger [2] and need not be reiterated here. For the calculation of the unresolved band envelopes, each rotational line was convoluted with an appropriate monochromator slit function.

In the case of the rotational temperature measurements in N_2 and N_2^+ for the high pressure Ne MHC we added a 1% admixture of N_2. Discharge currents varied between 0.5 and 3 mA. Fig. 1 shows a comparison of a measured spectrum of the (1,2) band of the N_2 second positive band recorded at a discharge current of 1 mA with a model spectrum corresponding to a rotational temperature of 375 K. The model spectrum and the measured spectrum were normalized to one

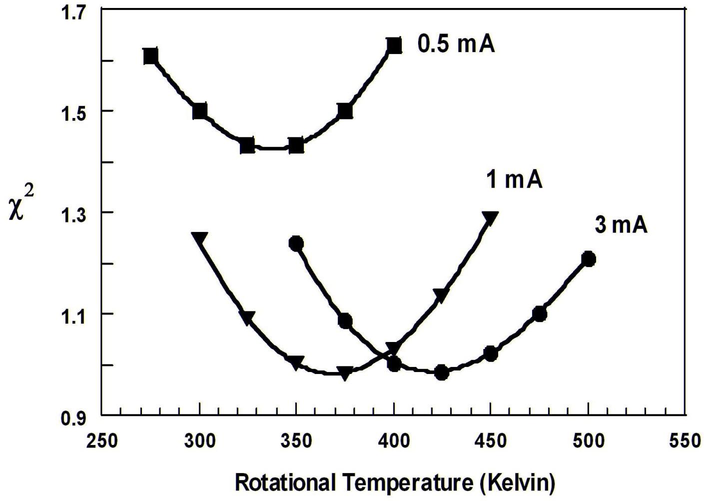

Fig. 2. χ^2 (see text for definition) as a function of the rotational temperature for the rotational analysis of the N_2 second positive band in a MHC discharge at three different discharge currents.

another at the position of the maximum. The agreement between the 2 spectra is generally good except perhaps around 353 nm, which is the onset of the R-branch lines, where the calculated spectrum lies slightly above the measured data. Fig. 2 shows the result of a systematic statistical analysis of fits corresponding to various temperatures for discharge currents of 0.5 mA, 1 mA, and 3 mA. We plot the relative "chi-squared", χ^2, as a function of the selected T_R value for the fit [the relative χ^2 is defined as $\chi^2 = \Sigma\,[y_i - y(x_i;T_R)]^2$ ($i = 1, \ldots, N$) where y_i denotes the measured intensity at the i-th wavelength position, $y(x_i;T_R)$ refers to the calculated intensity at the same wavelength position for a fixed T_R, and the sum extends over all data points from 1 to N]. In all three cases, the curves display clear minima at temperatures of 375 K for a discharge current of 1 mA, at 425 K for 3 mA, and between 325 K and 350 K for a discharge current of 0.5 mA. The shape of the χ^2 curves also suggests that the determination of the rotational temperatures can be accomplished to better than $\pm 10\%$ (or about ± 25 K). A similar rotational analysis was carried out for the N_2^+ first negative system. Fig. 3 shows the result of the statistical analysis for N_2^+ at the same three discharge currents of 0.5 mA, 1 mA, and 3 mA as before. Again, the three χ^2 curves display clear minima. However, the rotational temperatures are much higher compared to N_2 with values of 875 K, 1 050 K, and 925 K (with a similar accuracy of about ± 25 K) for currents of 0.5 mA, 1 mA, and 3 mA, respectively. The rotational temperatures obtained for N_2^+ are thus higher by about 600 K compared to the rotational temperatures obtained for N_2 and do not seem to increase monotonically with increasing discharge current.

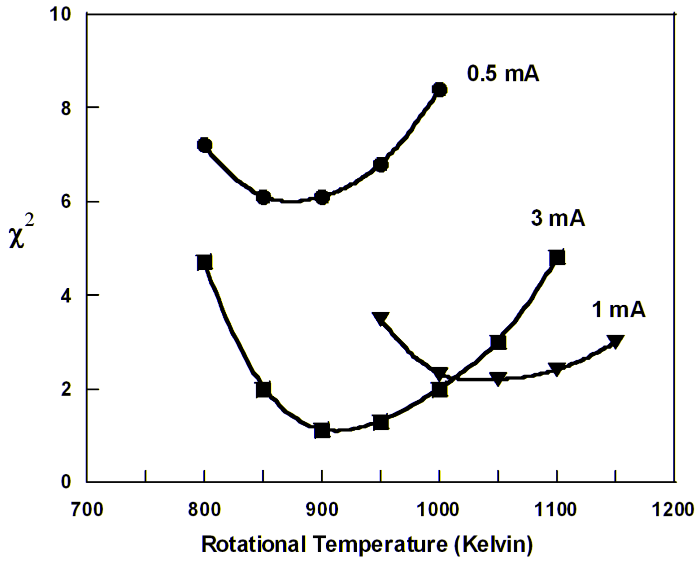

Fig. 3. χ^2 (see text for definition) as a function of rotational temperature for the rotational analysis of the N_2^+ first negative band in a MHC discharge at three different discharge currents.

The significant difference in the rotational temperatures obtained for N_2 and N_2^+ in high-pressure MHC discharges in Ne with 1% N_2 can be attributed to the different pathways involved in the excitation and relaxation of the two emitting states. The main route to the formation excited $N_2(C\ ^3\Pi_u,\ \nu=1)$ molecules which give rise to the observed (1,2) band of the second positive system of N_2 is electron impact from the N_2 ground state and from metastable N_2 states. The N_2 molecules in the ground state and in the metastable states are in equilibrium with the gas kinetic temperature after a few collisions with the Ne atoms. This is a fast process due to the high collision frequencies at high pressure. The rotational relaxation number Z_{rot}, which characterizes the exchange of rotational and translational energies of the plasma species and which is given by the ratio of the rotational relaxation time to the collision time, has been measured and calculated in Ne/N_2 mixtures to be around 2 [16] and the rotational relaxation time has a value of about 1 ns under the operating conditions in our MHC discharge. The apparent radiative lifetime of the emitting C-state is about 18 ns (which is a factor of 2 less than the natural radiative lifetime of 37 ns because of collisonal quenching). Thus, the effective lifetime of the emitting state is about 18 times longer than the rotational relaxation time and the emitting state is in thermal equilibrium with the bulk gas. This means that the rotational temperatures obtained from the analysis of the

N_2 second positive band under these conditions can be interpreted as the gas kinetic temperatures in the plasma.

By contrast, the formation of N_2^+(B $^2\Sigma_u^+$, ν=1) ions proceeds via electron impact ionization of N_2, via charge exchange of N_2 with Ne^+ and Ne_2^+ ions, and via a two-step process involving Penning ionization of N_2 by Ne metastables into the ground state of the ion and the lower-lying A-state followed by electron impact excitation to the B-state. In addition, three-body collisions involving Ne_2^+, N_2, and Ne can also result in the formation of N_2^+(B) ions, albeit with a much smaller rate constant [17]. Ne_2^+ are formed in appreciable quantities in discharges in high-pressure Ne as they are precursors to the formation of Ne_2^* excimers (see e.g. Refs. [5] and [18]). Khacef et al. [17] summarized various reaction channels in Ne/N_2 discharges involving collisions between Ne^+ or Ne_2^+ with N_2 leading to the formation of N_2^+(B) ions and provided rate constants. In our 400 Torr Ne MHC discharge with 1% N_2 admixture, two-body collisions are much more important in the formation of N_2^+(B) ions than three-body collisions and collisions between vibrationally excited Ne_2^+ ions and N_2 are the dominant reaction pathway. Depending on the amount of vibrational excitation, the Ne_2^+ ions carry between 18.9 eV and 20.3 eV energy (using the two Ne atoms with zero translational kinetic energy as the "zero" of the energy sale). This energy is marginally higher than the minimum energy (18.75 eV) required to form N_2^+(B) ions. Therefore, the higher rotational temperatures obtained from the rotational analysis of the N_2^+ first negative band in our MHC discharge are likely the result of rotational heating of the N_2^+(B) ions formed in charge exchange collisions between N_2 and Ne_2^+ ions in conjunction with the fact that the rotationally excited N_2^+(B) ions have insufficient time to relax prior to emission. The N_2^+(B) ions are efficiently quenched via collisions with N_2 and Ne [19, 20] and via dissociative recombination [21]. For the operating pressure of 400 Torr in our discharge and a 1% N_2 admixture we estimate quenching lifetimes of the excited N_2^+(B) ions of about 13 ns (collisions with N_2), 3.5 ns (collisions with Ne), and 20 ns (dissociative recombination for an electron density of 5×10^{14} cm^{-3}). Thus, binary quenching collisions shorten the natural radiative lifetime of the N_2^+(B) ions of 55 ns to an effective radiative lifetime of about 2 ns (which is still an upper limit as it does not take into account other binary quenching processes with e.g. Ne_2^* excimers and metastables and three-body quenching collisions). When compared with the rotational relaxation time of about 1 ns, it is apparent that the excited N_2^+(B) ions will not achieve thermal equilibrium with the bulk gas, but rather retain much of their rotational excitation prior to emission. This explains the much higher rotational temperatures obtained for N_2^+ ions compared to the N_2 molecules.

We limited the rotational temperature measurements in our high pressure CPE discharge in ambient air to the analysis of the N_2 second positive band system. A typical emission spectrum of the (0,1) band of the N_2 second positive system near 357 nm from the CPE discharge is shown in Fig. 4. The measured spectrum is compared with calculated spectra corresponding to three temperatures, 350 K, 545 K, and 650 K. The calculated curve corresponding to T_R = 545 K represents the best fit to the measured spectrum. The spectrum corresponding to T_R = 350 K clearly underestimates the short wavelength part of the band, whereas the spectrum corresponding to T_R = 650 K overestimates the same part of the band. The "best fit" corresponding to a T_R value of 545 K is the result of a careful statistical analysis of the comparison between the measured and the calculated spectra and has been determined with a margin of un certainty of about ±20 K. The rotational temperature obtained in the CPE discharge in ambient air for N_2 is significantly higher that the rotational N_2 temperatures obtained in MHC discharges in 400 Torr

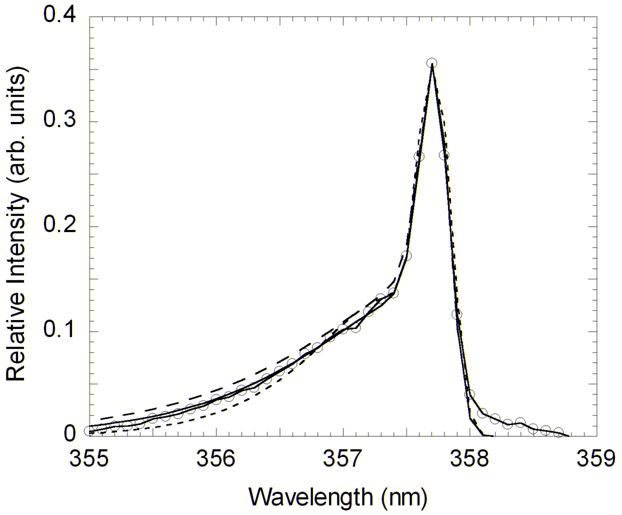

Fig. 4. Comparison of a measured spectrum of the (0,1) band of the N_2 second positive band recorded at a discharge current of 5.3 mA per capillary in an atmospheric-pressure CPE discharge in ambient air with model spectrum corresponding to a various rotational temperatures. The experimental data points are shown as the open circles connected by a thin solid line. The thick solid line represents the model spectrum corresponding to a T_R value of 545 K. The two dashed lines correspond to calculated spectra corresponding to T_R values of respectively 650 K (long dashed line) and 350 K (short dashed line).

Ne with 1% N_2. Two factors may contribute to the higher rotational temperatures in the case of the CPE discharge. First of all, the optical set-up in the case of the CPE discharge is such that only radiation from inside the capillary, which is the region of highest plasma density and highest gas temperature in this discharge type [6] is focused onto the entrance slit of the monochromator and analyzed in terms of its rotational temperature. Secondly, the quenching of the excited N_2(C) molecules is more effective at atmospheric pressure in the CPE discharge (as compared to 400 Torr in the MHC discharge) and may shorten the effective lifetime of the excited molecules to a value that is closer to the 1 ns rotational relaxation time. Thus, any rotational excitation that may have been imparted on the N_2(C) molecules upon their formation may be partially retained and the N_2(C) molecules may not be in thermal equilibrium with the bulk gas in the plasma.

4 Summary

We carried out rotational temperature measurements in high-pressure (400 Torr) microhollow cathode (MHC) discharges in Ne with a trace admixture of N_2 using the unresolved N_2 second positive band and the N_2^+ first negative band. The rotational temperatures obtained from the analysis of the N_2 band system are slightly above room temperature (around 400 K), increase with increasing discharge current, and may be interpreted as the gas kinetic temperature in the MHC discharge plasma under these operating conditions. By contrast, the analysis of the N_2^+ band system yielded rotational temperatures of more than 900 K, which are the result of collisional reaction kinetic processes leading to the formation of rotationally excited N_2^+ ions. The effective lifetime of these ions is comparable to the rotational relaxation time, so that the ions retain much of their rotational excitation prior to emission and are not in thermal equilibrium with the bulk gas. We also carried out a rotational analysis of the unresolved N_2 second positive system emitted by an atmospheric-pressure capillary plasma electrode (CPE) discharge in ambient air and measured a temperature of 545 K. This temperature may be close to the gas kinetic temperature as the rotational analysis utilized N_2 emissions from inside the capillary which is the region of highest plasma density and highest gas temperature in a CPE discharge.

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