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Measurement of Neutral Gas Temperature in a 13.56 MHz Inductively Coupled Plasma

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Abstract. Measuring the temperature of neutrals in inductively coupled plasmas (ICP) is important as heating of neutral particles will influence plasma characteristics such as the spatial distributions of plasma density and electron temperature. Neutral gas temperatures were deduced using a non-invasive technique that combines gas actinometry, optical emission spectroscopy and simulation which is described here. Argon gas temperature in a 13.56 MHz ICP were found to fall within the range of 500 - 800 K for input power of 140 - 200 W and pressure of 0.05 - 0.2 mbar. Comparing spectrometers with 0.2 nm and 0.5 nm resolution, improved fitting sensitivity was observed for the 0.2 nm resolution.

Keywords: actinometry optical emission spectroscopy, inductively coupled plasma, neutral gas temperature

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INTRODUCTION

Advances in modern emission spectrometry diagnostics and fiber technology has allowed for actinometry optical emission spectroscopy (AOES) to be a viable alternative in determining the temperature of the neutral particles in vacuum plasma systems. High resolution portable spectrometers offer the flexibility of being able to measure the full range of the plasma emission spectrum; from which various plasma properties can be deduced with ease of use and at the fraction of the cost. Results of neutral temperature measurement from these spectrometers using AOES has been shown to be of comparable accuracy with measurements made using other established laser spectroscopy techniques [1, 2].

The AOES method for determination of neutral gas temperature, \( T_n \), involves the usage of a probe gas or actinometer which is typically a molecular gas (i.e., nitrogen gas or oxygen gas) with well-defined theoretical vibro-rotational spectra. The probe gas is seeded in miniscule amounts (<5%) into the plasma to avoid from influencing other plasma properties. The emission spectrum of the resultant plasma is recorded and the peaks attributed to the ground state transitions of the molecular gas are analyzed. A peak belonging to a single vibro-rotational transition is chosen for determination of \( T_n \). Peak broadening as a result of the increase in rotational temperature of the nitrogen molecules in transition, \( T_{rot} \), is taken to be analogous to the increase of ground state nitrogen gas temperature, \( T_{N_2} \); which is assumed to be in equilibrium with the neutral gas temperature, \( T_n \), i.e., \( T_{rot} \approx T_{N_2} \approx T_n \) [3]. A broadened synthetic peak calculated from spectroscopic theory and instrumental broadening is fitted with the measured peak at different rotational temperatures and the best fitted temperature is determined as the neutral gas temperature of the plasma.

The temperature of the neutral gas in the inductive coupled plasma (ICP) has been reported to influence various important plasma characteristics including particle transport dynamics, power deposition, mode transition and field-plasma interaction. Much like the electron density and electron temperature, knowledge of the neutral gas temperature is important in temperature sensitive applications such as biomedical treatment, material deposition and reactive ion etching. In this work the methodology involved in obtaining the neutral gas temperature, \( T_n \) of argon plasma from AOES with a nitrogen gas actinometer is discussed. Measurement of \( T_n \) at the argon pressure range of 0.05 - 0.2 mbar using: (a) A 0.5 nm resolution spectrometer at increasing r.f. power (140 - 200 W) and (b) 0.2 nm and 0.5 nm resolution spectrometers at 200 W r.f. power are also shown.
METHODOLOGY

FIGURE 1. A 13.56 MHz ICP system setup for AOES measurement

Experimental Setup

A 13.56 MHz 6-turn planar coil ICP system set up for AOES measurement is shown in Fig. 1. Provisions have been made to include an additional gas inlet to provide nitrogen gas which is seeded in miniscule amounts (<5%). The amount of seeded probe gas needs not to be exact but only to be sufficient in order to obtain a nitrogen spectrum of observable intensity. At higher pressures (>0.2 mbar), ~1% of nitrogen would be adequate to give measurable peaks. A optical fiber probe with collimating (convex) lens assembly is connected to the Ocean Optics HR4000 spectrometer and aligned using an alignment laser before measurement. Both collimating lens and probe alignment are important so that the probe only collects the radial, line averaged emission from the plasma which would represent emission measurement at a certain axial distance from a reference point. As with most spectrometry measurements, the exact intensity of the spectrum is not as vital as the relative intensities of the emission peaks. In this experiment, the fiber was set up to measure the line emission at the height of 3.2 cm from the dielectric plate. This would translate as measuring the spatially averaged neutral gas temperature of the plasma, at 3.2 cm distance from the dielectric plate.

Measurement of Emission Spectra

The emission spectra of the nitrogen seeded argon plasma was measured for the pressure range of 0.05-0.2 mbar at 140-200 W r.f. power the 0.5 nm resolution HR4000 spectrometer and at 200 W r.f. power using another 0.2 nm resolution HR4000 spectrometer for comparison. This is followed by the selection of a suitable vibro-rotational peak that would be used to determine the neutral gas temperature. In many established works, the vibro-rotational peaks from second positive system of molecular nitrogen (N$_2^3\Sigma^+_u$-$N_2^3\Sigma^+_g$) are typically used for AOES with argon plasma [3, 4, 5]. In this system, it is observed that the measured (0-2) vibro-rotational transition peak has no overlap, is distortion free (from other transitions) and is of resolvable intensity; thus, the (0-2) peak was chosen for fitting with the theoretical synthetic spectra.

Calculation of Discrete Synthetic Spectra

*The following summarizes the important equations used in computation. Further details on the calculations of terms and determination of degenerate states (including the complete set of equations and constants) can be found in Refs. 3, 6 and 7.
Vibro-rotational spectroscopic theory for AOES involves the calculation of discrete spectral lines which constitute a synthetic spectrum that will be fitted with the observed experimental peak. In order to determine the spectral lines, the spectral line positions and line intensities of contributive rotational transitions and line degeneracies for the (0-2) \( \text{N}_2^3\Pi_u^\text{vib} - \text{N}_2^3\Pi_g^\text{vib} \) vibrational peak are required. The spectral line position, \( \lambda_i \), is given by,

\[
\lambda_i (v', v'', J', J'') = \frac{1}{v_i (v', v'', J', J'') \cdot n_{\text{air}}}.
\]

Here, \( v \) represents the vibrational quantum number, \( J \), the rotational quantum number, \( T_{\text{rot}} \), the rotational temperature (K) and ' the upper transition and '' lower transition states respectively; thus, \( v' \) and \( v'' \) denotes the vibrational quantum number in the upper and lower transition states.

\[

\nu_i = \frac{(T_{\text{elec}} + G(v) - F_v(J') - F_v(J''))}{h c}.
\]

where, \( T_{\text{elec}}, G(v) \) and \( F_v(J) \) are the electronic, vibrational and rotational terms, respectively in m\(^{-1}\). The subscript \( i \) represents the 3 allowed transition branches for the rotational states namely, \( R(J' - J'' = 1), Q(J' = J'' = 0) \) and \( P(J' - J'' = -1) \). For the presented case, calculation of rotational lines was made for the rotational quantum numbers, \( J = 0 \rightarrow 50 \). In addition to the rotational lines generated by the allowed transition branches, further rotational transitions from degenerate states also give significant contribution to the observed spectrum (for higher resolution spectrometers, inclusion of degenerate states increases fitting accuracy). According to Hund’s case (a), the \( ^3\Pi \) state can be split to 3 degenerate states at which rotational transitions would occur, which is notated by \( ^3\Pi_0 \rightarrow ^3\Pi_1 \) and \( ^3\Pi_2 \). From the various possible transitions, three major degenerate transitions were included in calculation, i.e., \( ^3\Pi_0 \rightarrow ^3\Pi_1 \rightarrow ^3\Pi_1 \) and \( ^3\Pi_2 \rightarrow ^3\Pi_2 \).

The spectral line intensity, \( I_{\text{emiss}} \) is given by,

\[
I_{\text{emiss}} (J', T_{\text{rot}}) = C \cdot S_J \cdot \exp \left( -\frac{F_v(J)(J'+1)hc}{k T_{\text{rot}}} \right).
\]

\( C \) is a constant dependent on spectroscopic parameters, \( S_J \) is the line strength, \( h \) is Planck’s constant (Js\(^{-1}\)), \( c \) is the speed of light (cms\(^{-1}\)), \( F_v(J') \) is the upper state rotational term (cm\(^{-1}\)), \( k \) is the Boltzmann constant (JK\(^{-1}\)) and \( T_{\text{rot}} \) is the rotational temperature of nitrogen (K). The exact value of constant \( C \) is not important for this analysis as only the relative intensities are used for comparison (i.e., \( C \) is taken as 1). \( S_J \) is a function of the rotational quantum number, \( J \) using the Honl-London formulae (which also takes into account state degeneracy). An example of calculated discrete synthetic spectra is shown in Fig. 2 (a). For the selected (0-2) vibro-rotational peak a total of 328 discrete lines were considered.

**FIGURE 2.** (a) The discrete synthetic spectra at \( T_{\text{rot}} = 550 \) K (b) The convolved synthetic spectra at \( T_{\text{rot}} = 550 \) K at 0.5 nm instrumental broadening.
Convolution of Instrumental Broadening into Synthetic Spectra

The discrete synthetic spectra alone would not be a proper representation of the experimental spectrum. Usually, measured emissions are subject to spectral broadening due to the mechanical limitations of the instrument, i.e., diffraction by the spectrometer grating, dispersion by mirrors, CCD resolution, alignment and refractive errors. The resultant sum of these instrumental losses is known as instrumental broadening. The amount of instrumental broadening of the selected vibro-rotational peak can be estimated by taking the single line emission broadening of a standard mercury lamp which is closest to the peak. In the presented case, broadening of the 435.8 nm mercury line emission peak was used. To apply instrumental broadening into the synthetic spectra, the 435.8 nm mercury peak is first fitted to a spectroscopic function. The most widely used function for these fittings is the Voigt profile; due to the inclusion of both Lorentzian and Gaussian broadening mechanisms in its parameters. Once the peak is fitted, the resultant function serves as representative of instrumental broadening of every rotational emission lines in the discrete synthetic spectrum. Incorporation of the fitted Voigt broadening function into the discrete synthetic spectra is done by using the method of convolution, i.e.,

\[
I_{\text{conv}}(\lambda) = \int_{-\infty}^{\infty} I(\lambda') \cdot f(\lambda - \lambda') d\lambda' .
\]

\(I_{\text{conv}}(\lambda)\) is the convoluted intensity at wavelength \(\lambda\), \(I(\lambda')\) is the intensity at the integration wavelength \(\lambda'\) and \(f(\lambda - \lambda')\) is the Voigt profile with peak wavelength \(\lambda\) at displacement \(\lambda'\). The result of convolution on the discrete synthetic spectra is demonstrated in Fig. 2 (b). The process of calculation and convolution of the synthetic spectra is done for predetermined rotational temperature, \(T_{\text{rot}}\) range and increment. In the presented case, temperatures from 250 - 1500 K were computed at increments of 50 K.

Statistical Analysis of Measured and Convolved Spectra

The final step in determining the neutral gas temperature with the AOES method is statistical fitting of the convolved synthetic spectra and measured spectrum. Before fitting, the data points of both the convolved synthetic spectra and measured spectrum should be normalized and interpolated such that the data points represent the central wavelength intensities of predetermined bins; with the bin size being the minimum resolution of the spectrometer. These bins are defined so that the deviation between theoretical and experimental values can be calculated and compared. Statistical fitting of the convolved synthetic peak and measured peak is done using the \(\chi^2\) analysis, i.e.,

\[
\chi^2 = \sum_{i=1}^{N} P(x - x_o)^2 \cdot \frac{1}{N(N-1)} .
\]

Here, \(N\) is the total number of bins, \(x\) is the measured spectrum intensity and \(x_o\) is the synthetic spectrum intensity at the same wavelength as \(x\). \(P\) is the probability (weight) of discrete lines in each bin which is given by,

\[
P = \frac{\text{Number of Discrete Lines in Bin}}{\text{Total Number of Discrete Lines in Spectra (328)}} .
\]

The \(\chi^2\) values are calculated for the convolved synthetic spectra of the rotational temperature range 250 - 1500 K. The rotational temperature, \(T_{\text{rot}}\) at which the \(\chi^2\) value is minimum, is taken as the estimated neutral gas temperature, \(T_n\) of the plasma.

RESULTS AND DISCUSSION

Fig. 3(a) shows the neutral gas temperature, \(T_n\) versus r.f. power, measured at 0.05 - 0.2 mbar argon pressure using a 0.5 nm spectrometer. From Fig. 3(a) it is seen that \(T_n\) gradually increases with r.f. power, due to the increased power deposition of the source into plasma particles. Deviation of the measured data at 140 W for 0.1
mbar argon pressure is due the point being close to the mode transition point [8]. However, more significant increase in $T_n$ is seen at the same measured power with increasing argon pressure. This can be explained as follows: As argon pressure is increased, the number of particles in the chamber increases. The frequency of collisions between particles increase and energy exchange between particles becomes more efficient [9]. The argon neutrals receive more kinetic energy from the plasma particles resulting in the observed increase of $T_n$. Differences in trends of $T_n$ with argon pressure are also seen from measurements made by different resolution spectrometers at 200 W (Fig. 3(b)). The better defined increase in $T_n$ shown by measurements made with the 0.2 nm spectrometer is due to the increased resolvability of the finer variations in the (0-2) vibro-rotational peak, thus, improving fitting sensitivity. The standard deviation of $T_n$ were between 4-5 % for the 0.2 nm spectrometer and 2-7% for the 0.5 nm spectrometer.

![Graphs](image)

**FIGURE 3.** (a) Neutral gas temperature, $T_n$ (K) versus r.f power (W) at different argon pressures for the 0.5 nm spectrometer. (b) Neutral gas temperature, $T_n$ (K) versus argon pressure (mbar) in logarithmic scale for the 0.2 nm and 0.5 nm spectrometers at 200 W r.f. power.

**CONCLUSION**

The technique for measurement using the AOES method has been briefly detailed. The measured $T_n$ values were comparable to those obtained in similar ICP systems as reported by Li et al. [10] using the AOES method and Shimada [3] using the AOES and LIF methods. Measurement with a 0.5 nm resolution spectrometer showed gradual increase in neutral gas temperature, $T_n$ with r.f. power and more significant increase with argon pressure. The increase of $T_n$ with r.f. power is attributed to the increased power deposition of the source into the plasma particles whereas, the increase with argon pressure is attributed to the increased energy transfer efficiency between the plasma particles and the neutrals. Comparison of $T_n$ measurements with argon pressure between 0.2 nm and 0.5 nm resolution spectrometers showed better fitting sensitivity with the 0.2 nm spectrometer.

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