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Plasma diagnostics of a DC glow discharge using Tuneable Diode Laser Absorption Spectroscopy

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Abstract

The behaviour of plasmas is influenced by thermodynamic quantities such as gas and electron temperatures and their corresponding number densities. For applications such as thin-film deposition, material synthesis, sputter coating, spark plugs, and spacecraft thrusters that depend upon plasma processes it is of practical interest to quantify these physical properties in order to optimise their performance and efficiency. In this work, time-resolved tuneable diode laser absorption spectroscopy (TDLAS) is used to characterise an Argon glow discharge in a Dynavac pulsed DC sputter coater. This is achieved by current scanning a vertical-cavity surface-emitting laser (VCSEL) at a known neutral Argon transition wavelength of 794.8 nm that corresponds to an energy level transition from the $3s^2 3p^5 4s$ metastable state to the excited upper state $3s^2 3p^5 4p$ [4]. The pressure and current conditions of the sputter coater are kept constant at 11 Pa and 24 mA respectively during the experiment. A Doppler-broadened absorption profile is assumed from which both time-resolved temperature and number density are calculated. It was found that the plasma temperature is constant to within measurement uncertainty for a given duty cycle, while the number density variation over a cycle follows the voltage characteristics of the device.

Introduction

The behaviour of plasmas is determined by physical properties such as gas and electron temperatures and densities. Applications such as thin-film deposition, material synthesis, sputter coaters, spark plugs and thrusters rely upon plasma processes. Therefore it is of practical interest to quantify these physical properties so that the application can be made to perform optimally. In the case of a sputter coater, this would mean that the rate of deposition was maximised. It is beneficial if the diagnostic technique is inexpensive, easy to set up and does not physically interfere with the plasma deposition process. Unlike Langmuir probes that use one or more electrodes to determine electron and ion temperatures and densities, optical diagnostic techniques such as optical emission spectroscopy (OES) and absorption spectroscopy (AS) make their measurements non-intrusively. In comparison to Langmuir probes, OES and AS can provide better temporal resolution of the plasma properties and are not susceptible to contamination by sputtered species and consequently have been widely used in plasma diagnostics [5, 1].
The following paper describes the use of tunable diode laser absorption spectroscopy (TDLAS) to determine the temperature and number density of the ionisation gas in a commercial sputter coater. TDLAS is a well-established method and is used as a diagnostic tool in applications such as combustion [3], flow characterisation [6] and soil and water analysis [2]. In comparison with OES, TDLAS can provide superior spectral resolution when a narrow-linewidth laser source is used, and accesses different atomic or molecular states that can be unavailable to emission based systems, most importantly including the ground state. Absorption spectroscopy is based on the principle that relates the amount of light absorbed to the properties of the absorbing medium. This is described by the Beer-Lambert law illustrated in figure 1.

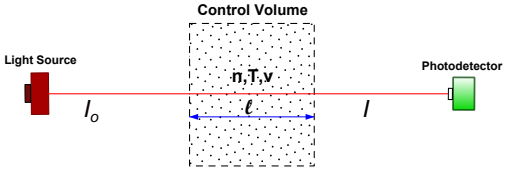

Figure 1. Simplified absorption system diagram

The Beer-Lambert law is given by the following correlation:

$$k_0 l = \ln \frac{I_0}{I}, \tag{1}$$

where

l is the absorbing path length
I_0 is the total irradiance of the light source
I is the transmitted irradiance, and
k_0 is the absorption coefficient given by,

$$k_0 = k_0(n, T, v). \tag{2}$$

Here n, T, v correspond to the number density, temperature and velocity of the given species.

The aim of this experiment is to determine the temperature and number density of metastable Argon in a pulsed DC glow plasma in the Dynavac SC 150 sputter coater using TDLAS. This is achieved by scanning a diode laser at the transition wavelength of 794.818 nm that corresponds to an energy transition between the Ar I $3s^2 3p^5 (^2P^o_{1/2}) 4s$ metastable state and an excited upper state $3s^2 3p^5 (^2P^o_{1/2}) 4p$. The translational temperature is determined from the Doppler width of the transition, while number density is determined from the integrated absorbance signal.

Experimental Arrangement

Absorption measurements are conducted downstream of the sputter coater as shown in the schematic in figure 2. The laser beam path originating from a Vixar single-mode VCSEL mounted on a Thorlabs LDM21 Laser Diode Mount is tuned to a transition wavelength 795 nm using a Thorlabs LDC 200C

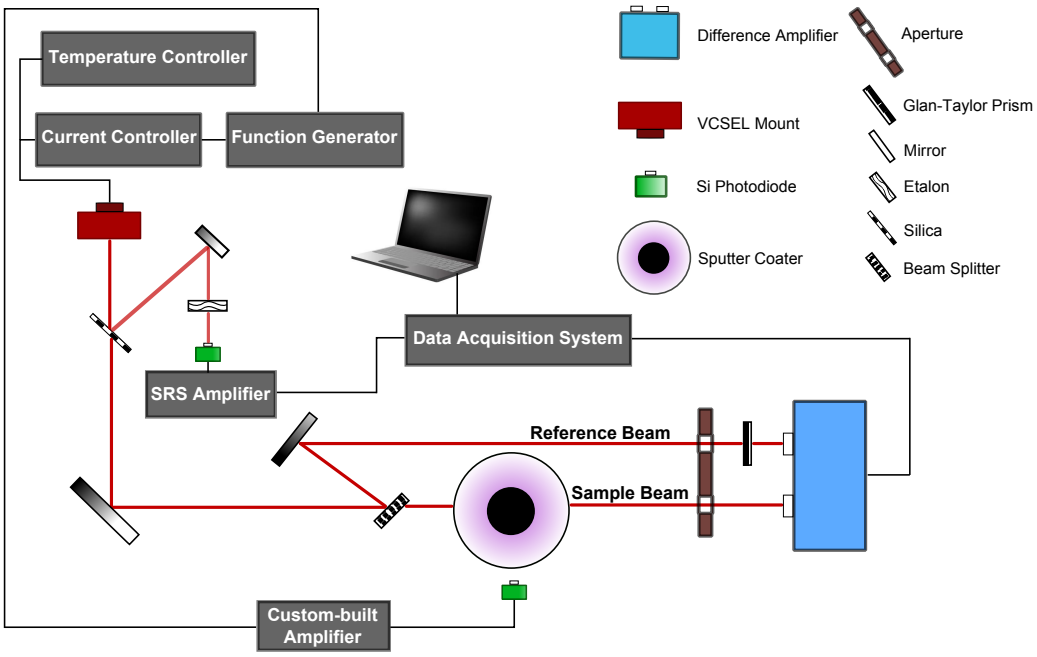

Figure 2. Schematic of the experimental setup

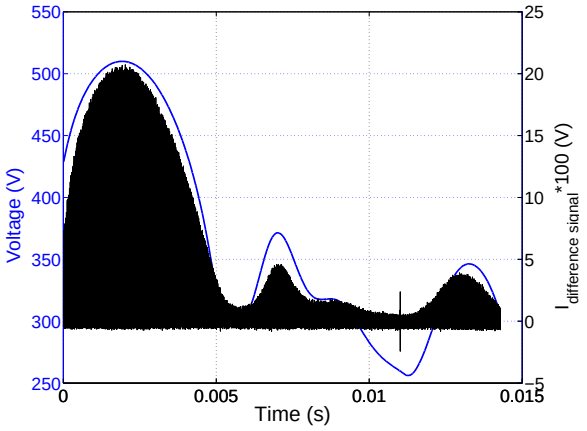

Figure 3. Voltage characteristics of the Dynavac sputter coater and the corresponding difference signal obtained from the difference amplifier (scaled 100 times)

VCSEL current controller and Thorlabs TED 200 temperature Controller. The current is modulated with a ramp waveform at 50 kHz using a Agilent 33220A 20 MHz function generator with a 30 mV amplitude. The function generator was operated in burst mode in order to capture the difference signal over the 50 Hz cycle of the device. The beam is split into reference (I_0) and sample beams (I) using a 70:30 beam splitter. An aperture screen with two 2 mm diameter slits for the beams is placed before the difference amplifier to block out the majority of the stray light from the plasma emission. A Glan-Taylor prism is used to adjust the intensity of the reference beam such that the output of the difference amplifier is zero or 'balanced' when the plasma is off. A New Focus 10 MHz Adjustable Balanced Photoreceiver (referred to as difference amplifier hereafter) is used to detect reference and sample beams and produces an amplified signal proportional to the difference between these two signals. The reference beam is recorded separately by blocking the sample beam. The pulsed DC characteristics of the sputter coater made it necessary to synchronise the function generator with the temporally varying 50 Hz device voltage in order to obtain a synchronised signal from the difference amplifier. It was found that the amplitude of the emission signal from the plasma followed the same temporal trend as the pulsed DC voltage of the sputter coater. This feature was exploited by using the amplified output signal of the photodiode diode facing the sputter coater to externally trigger the function generator as shown in figure 2. The difference signal is a composite of several spectra varying in amplitude as a direct consequence of the voltage characteristics of the sputter coater, as shown in figure 3. Figure 4 is a small slice of the difference signal zoomed that reveals the individual spectrum. The red highlighted lines denote the start and end of the ramp waveform that is used to scan the VCSEL.

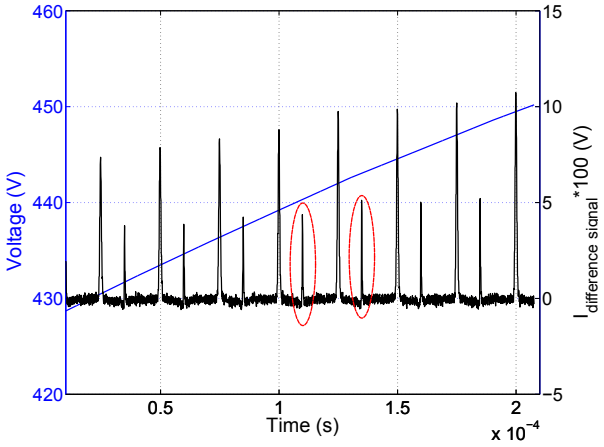

Figure 4. Spectra that constitute the difference signal

In order to acquire a wavelength calibration, a piece of fused silica is used to deflect some of the incident beam I_o onto a mirror which directs the beam onto an etalon producing etalon fringes that are detected by the silicon photodiode. Figure 5

shows the etalon fringes with a free spectral range of 0.25 cm^{-1} used to calibrate the absorbance signal. Each spectrum in the difference signal has a total of four etalons peaks which are interpolated along with the known position of the transition peak for a wavelength calibration. Finally, both the difference and etalon signals are recorded using a Cleverscope 10 MHz data acquisition unit. Data is recorded over a five minute period during which the plasma in the device is switched on. A total of 97 data sets were recorded at a sampling rate of approximately 4.6 MSPS which contained 476 spectra per scan.

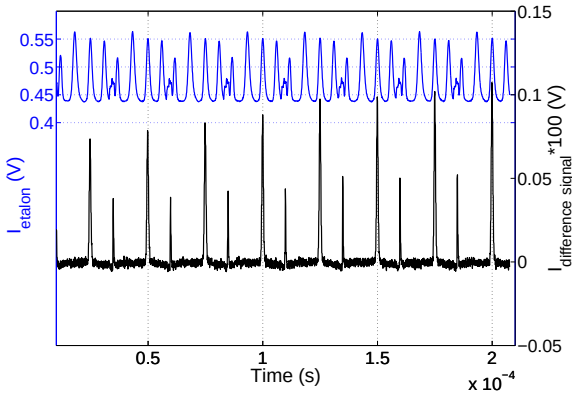

Figure 5. Etalon fringes (blue) used for wavelength calibration

Results and Discussion

The gas temperature is determined by calculating the full width at half maximum (FWHM) of a Gaussian profile fitted to the experimental data. Given the low operational pressure of 11 Pa it is reasonable to assume a Doppler-broadened profile since pressure broadening effects have a negligible impact on the absorption line. The plasma gas temperature is calculated using the following relation:

$$T_{gas} = \frac{m_{Ar}c^2}{8k\ln(2)}\left(\frac{\delta\nu_D}{\nu_0}\right)^2, \tag{3}$$

where

T_{gas} is the gas temperature in K
m_{Ar} is the mass of Argon in kg
c is the speed of light in m/s
$\delta\nu_D$ is the Doppler width or FWHM in cm^{-1}
ν_0 is the line center frequency in cm^{-1}
k is the Boltzmann constant in J/K

For a normally distributed Gauss profile, the FWHM is given by:

$$\delta\nu_D = 2\sqrt{2\ln(2)}\sigma_{Gauss}, \tag{4}$$

where σ_{Gauss} is the standard deviation of the fitted Gauss profile

The Gaussian parameters σ_{Gauss}, μ_{Gauss}, ν_0 and amplitude A of the theoretical absorbance profile were calculated by performing a least squares fit to a quadratic function which is obtained by taking the natural logarithm of the Gaussian function given by:

$$f(x) = A\exp\left(\frac{-(x-\mu)^2}{2\sigma^2}\right) \tag{5}$$

These parameters are then used to fit the absorption data as shown in figure 6. As shown in equation 3, the gas temperature is directly proportional to the square of the FWHM which means a relatively small under or over prediction of the Gaussian fit to the experimental data can result in significant errors in the temperature values.

An example of a fitted Gauss profile is shown in figure 6. It should be noted that the spectrum in figure 6 is averaged over 97 data sets. This was done to improve the quality of the fit to the fitted Gaussian profiles which would otherwise resulted in over predictions of gas temperatures. The deviations between the experimental data shown in figure 6 and the theoretical line fit is shown in figure 7 with a maximum residual error of 1.1%.

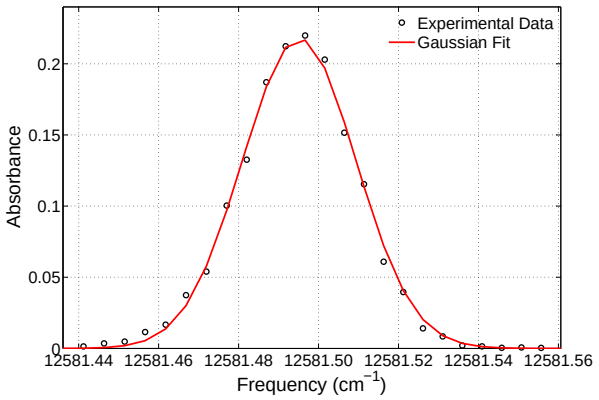

Figure 6. Averaged absorbance spectrum

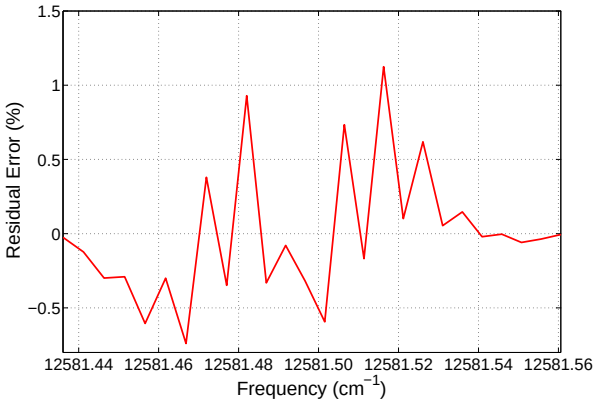

Figure 7. Residual error between experimental and theoretical absorption profiles

The number density of the metastable Argon was calculated using the equation found in [5]:

$$n_{Ar} = \frac{k_0\delta\nu_D}{8.25\times10^{-13}f}, \tag{6}$$

where

n_{Ar} is the number density of Argon in cm^{-3}
k_0 is the peak absorption coefficient in cm^{-1}
$\delta\nu_D$ is the Doppler width or FWHM in cm^{-1}
f is the oscillator strength which is related to the transition probability

k_0 is obtained by dividing the peak absorbance by the absorbing path length which is 7.5 cm. The value of the oscillator strength is 0.529 [4].

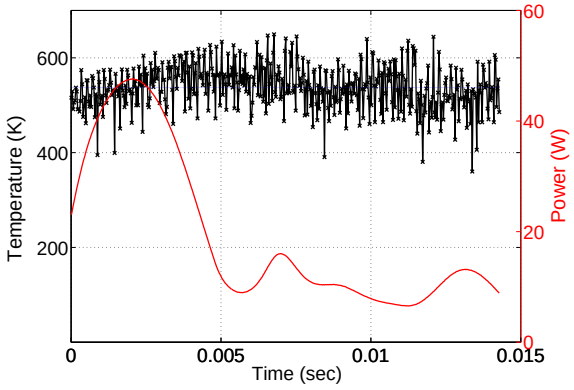

Figure 8. Temporal variation of temperature over a 50 Hz cycle and the corresponding power characteristics of the Dynavac sputter coater

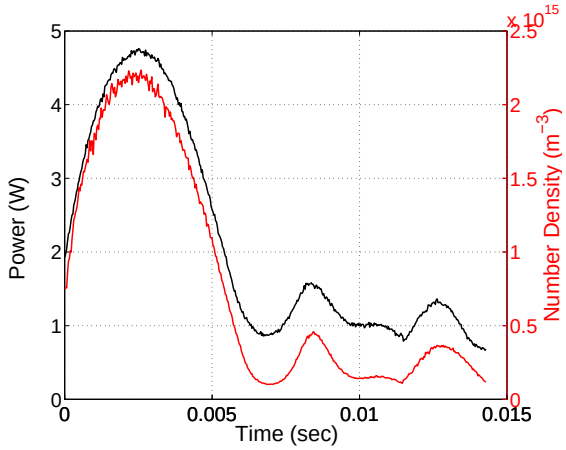

Figure 9. Temporal variation of the metastable Ar I number density and the corresponding power characteristics of the Dynavac sputter coater

Figure 8 shows the temporal variation of the gas temperature and power delivered to the plasma over a 50 Hz cycle. It should be noted that between 0.014–0.02 s the energy deposited into the gas does not result in any absorption and hence the temperature range is plotted prior to $t = 0.014$ s. The temperature variation is within 178 K from the mean value T_{mean} of 522 K with the maximum and minimum temperatures of 423 K and 600 K respectively. The rapid fluctuations in the inferred temperature are due to poorly fitted theoretical line curves which arise from a combination of under-sampled and/or poor signal-to-noise ratio (SNR) data. The results are surprising since a higher variation in gas temperature was expected considering the range of voltages used during a pulsed cycle. However the temporal variation of the absorption profile is directly proportional to the temporal variation of the metastable Argon species, as shown in figure 9.

The mean number density n_{mean} is 8×10^{14} m^{-3} with the maximum and minimum densities of 2.2×10^{15} m^{-3} and 1×10^{14} m^{-3} respectively.

Conclusions

Time resolved TDLAS was used to optically probe a pulsed DC glow discharge in a commercial sputter coater to determine the properties of the ionisation gas. Contrary to what was expected, it was found that the temporal variation of the absorption coefficient was a direct result of the temporally varying metastable Argon (Ar I) number density and not the gas temperature which remained more or less constant throughout the voltage pulse cycle. The mean translational temperature of the Ar I species was found to be constant at 522 K within the measurement uncertainty of approximately $\pm$ 90 K. The metastable Ar I species density varies with the voltage characteristic of the sputter coater with a n_{mean} of 8×10^{14} m^{-3}. The same technique can be used to determine time resolved properties such as deposition rates, number densities and temperature of the target material used in the vaporised thin film deposition process.

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