Laboratory Report

Microwave Plasma Chemical Vapor Deposition

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Objectives:

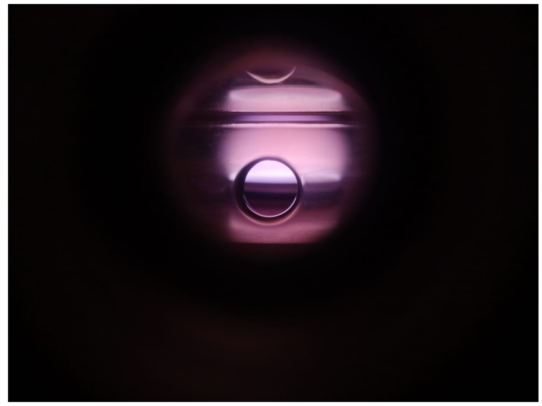
1. To study the Microwave Plasma Chemical Vapor Deposition (MPCVD) for plasma processing of carbon materials and the growth of high-quality single crystal diamond (SCD).
2. To study the Optical emission spectroscopy (OES) of hydrogen (H_2) plasma and identify the Balmer H_α and H_β lines.
3. To study the OES of hydrogen and methane (CH 4) plasma and identify the H_α , H_β , H_2 - Fulcher band, C_2 (swan bands, $\Delta\nu$ = -1, 0, 1), and CH band.
4. To estimate the electron temperature (T_e) using the H_α and H_β line intensities in H_2 - CH_4 plasma.

Theory:

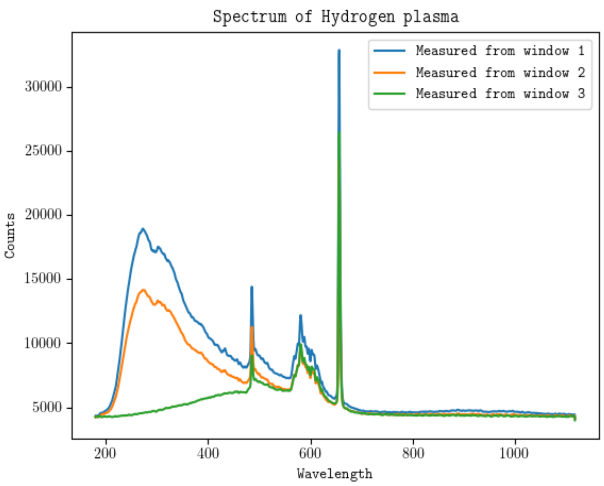
1. **MPCVD**
   MPCVD is a versatile and widely used technique for synthesizing high-quality diamond films and other carbon-based materials. This method uses the microwave energy to create a plasma environment, enabling the growth of diamond crystals with exceptional purity and quality. In MPCVD, a gas mixture typically composed of H_2 and a carbon source, such as CH_4 , is introduced into a vacuum chamber. Microwaves are then used to ionize the gases, creating a plasma that contains high-energy ions, radicals, and electrons. These reactive species interact with a substrate leading to the deposition of diamond films.
2. **Magnetron**
   A magnetron is a high-powered vacuum tube that generates microwaves using the interaction of a stream of electrons with a magnetic field. It consists of a cylindrical cathode placed at the center of an anode block, which typically has resonant cavities. When a high voltage is applied between the cathode and anode, electrons are emitted from the cathode and accelerated towards the anode. A strong magnetic field, perpendicular to the electric field, is applied, causing the electrons to spiral and create a circular motion around the cathode. As these electrons pass by the resonant cavities in the anode block, they induce high-frequency oscillations, generating microwaves.
3. **Vacuum system**
   The vacuum system is an integral part of the MPCVD technique, ensuring the creation of a controlled environment necessary for the deposition process. It allows precise control over gas phase composition, pressure, and temperature, which are essential for the growth of defect-free and high-purity materials. The rotary pump, also known as a roughing pump, is used to create a primary vacuum in the deposition chamber by reducing the pressure from atmospheric level to a medium vacuum range.
4. **Pyrometer**
   Digital pyrometers work on the principle of detecting infrared radiation emitted by an object. By measuring this radiation, the pyrometer can determine the temperature of the object without physically touching it. The lens used in the pyrometer collects the infrared radiation emitted by the object and focuses it onto the detector. The detector converts the focused infrared radiation into an electrical signal. The signal processor amplifies and processes the electrical signal to calculate the temperature.
5. **Optical emission spectroscopy**
   OES is based on the principle that atoms and ions emit light at characteristic wavelengths when they return to a lower energy state after being excited. The energy source, such as a plasma, provides sufficient energy to excite the atoms or ions in the sample. As these excited species return to their ground state, they emit light at specific wavelengths that correspond to the energy differences between the excited and ground states. The spectra of radical species such as $CH, C_2, and\ H$ (Balmer series) can be identified and analyzed using OES.

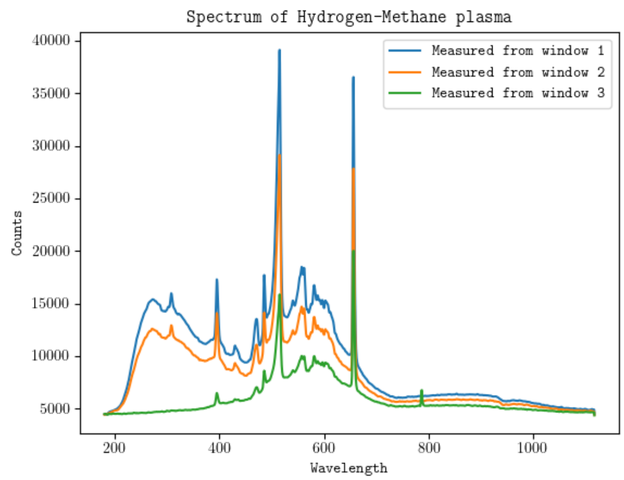
Observations:

Chamber pressure (Torr)	Plenum (Torr)	Microwave power (kW)	Temperature ($^\circ C$)	H2 flow (SCCM)	CH4 flow (SCCM)
60±0.1	20±1.5	2000± 10	475 ± 1	500±5	50±5

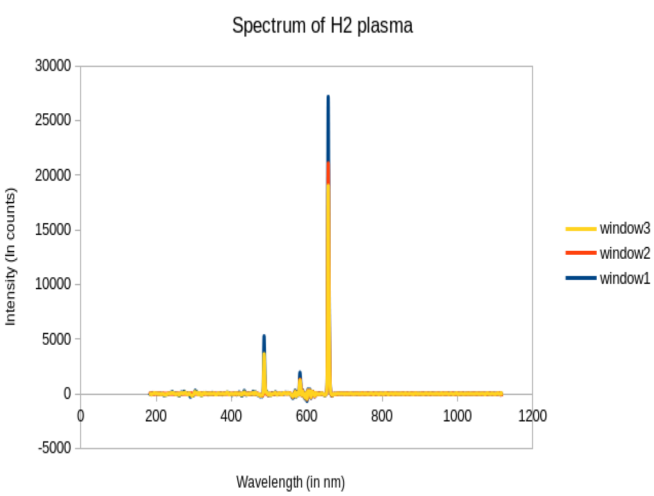


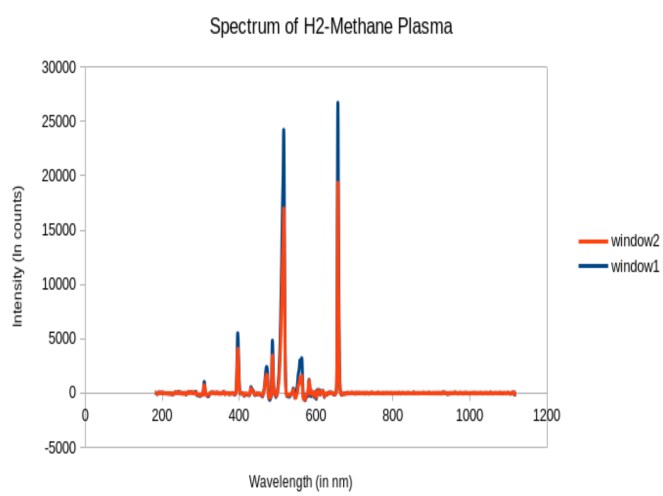
The left picture is the set up used for the experiment. The right picture is the H_2 plasma, the $H_2 - CH_4$ plasma has a green hue.



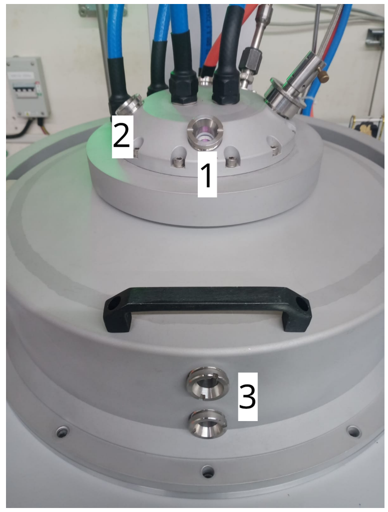


The left picture is the spectra of H_2 plasma obtained by keeping the spectrometer at different windows. The right picture is the spectra of $H_2 - CH_4$ plasma. Note that wavelengths are in nanometres





Both the spectrum after background correction in Origin Pro



The windows from which the spectrum was observed. Image modified with GIMP.

Analysis:

H_2 plasma:

We look at the graph and we notice a few observations

- We see a large presence of wavelengths at the range 200-400 nanometres. Even more surprising is that this is not recorded in the third window. A point to be noted is that in the third window, there is a quartz cylinder between the plasma and the spectrometer probe, which is not there between plasma and windows 1 and 2.
- The large count of the extra wavelengths (around 200-400 nm) in 1,2 can be attributed to the fact that, the probe also detects the emissions done by the heated plate, which emits between IR and visible region. As 3 is parallel to the plate, this is not observed there. This extra wavelength counts can be removed by baseline corrections.
- The low intensity is due to the fact that the quartz window shunts the intensity and also, 1 and 2 are closer to the plasma than 3.

We now measure the H_α, H_β lines from the combined spectrum

Band	Wavelength(nm)
H_α	656.5 $\pm$ 0.5
H_β	486.0 $\pm$ 0.5

$H_2 - CH_4$ plasma:

We note the same points discussed in the H_2 plasma. Without repeating that, we try to identify the H_α , H_β , H_2 - Fulcher band, C_2 swan bands, and CH band

Band	Wavelength(nm)
H_α	656.5$\pm$0.5
H_β	486.0$\pm$0.5
H_2	603.0 $\pm$ 0.5
C_2 $(\nu = -1)$	471.5 $\pm$ 0.5
C_2 $(\nu = 0)$	515.5 $\pm$ 0.5
C_2 $(\nu = 1)$	557.5(562) $\pm$ 0.5
CH	430.5 $\pm$ 0.5

As a final exercise, we try to calculate the electron temperature.

Electron temperature (T_e):

The electron temperature can be found out by

$$\frac{I(H_\beta)}{I(H_\alpha)} = N.exp(\frac{-\Delta E}{kT_e})$$

$$\implies T_e = -\frac{\Delta E}{k(ln(I(H_\beta) - ln(N) - ln(I(H_\alpha))}$$

$$\implies T_e = \frac{0.7}{ln(\frac{I_{H\alpha}}{I_{H\beta}}) - 0.78} eV$$

where

- ΔE≈0.65 eV, which is the energy difference between n=3→2 and n=4→2 levels.
- $I(H_\beta)$ and $I(H_\alpha)$ are the emission intensities of the H_α and H_β lines.
- N is a constant given by

$$N = \frac{A_{42}\nu_{42}g_4}{A_{32}\nu_{32}g_3}$$

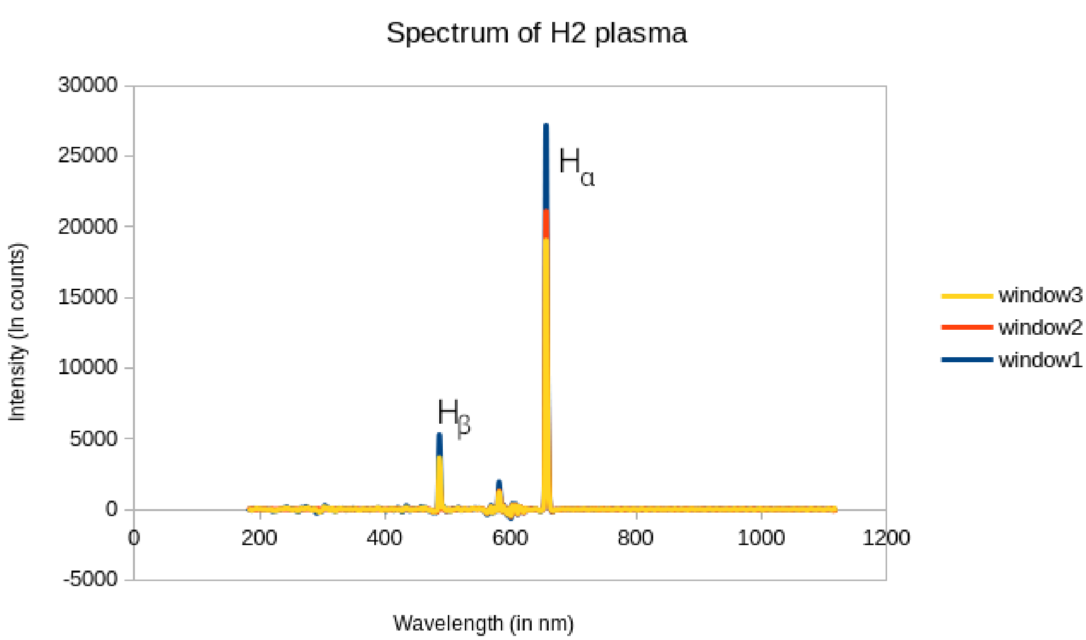
Where,

1. A_{42} and A_{32} are transition probabilities, where $A_{42} = 8.42 \times 10^6 s^{-1}$ for H_α and $A_{32} = 4.41 \times 10^7 s^{-1}$ for H_β
2. ν_{42} and ν_{32} are transition frequencies, where $\nu_{42} = 6.167 \times 10^{14} s^{-1}$ for H_α and $\nu_{32} = 4.568 \times 10^{14} s^{-1}$ for H_β
3. g_4 and g_3 are statistical weights, where $g_4 = 18$ for H_α and $g_3 = 32$ for H_β

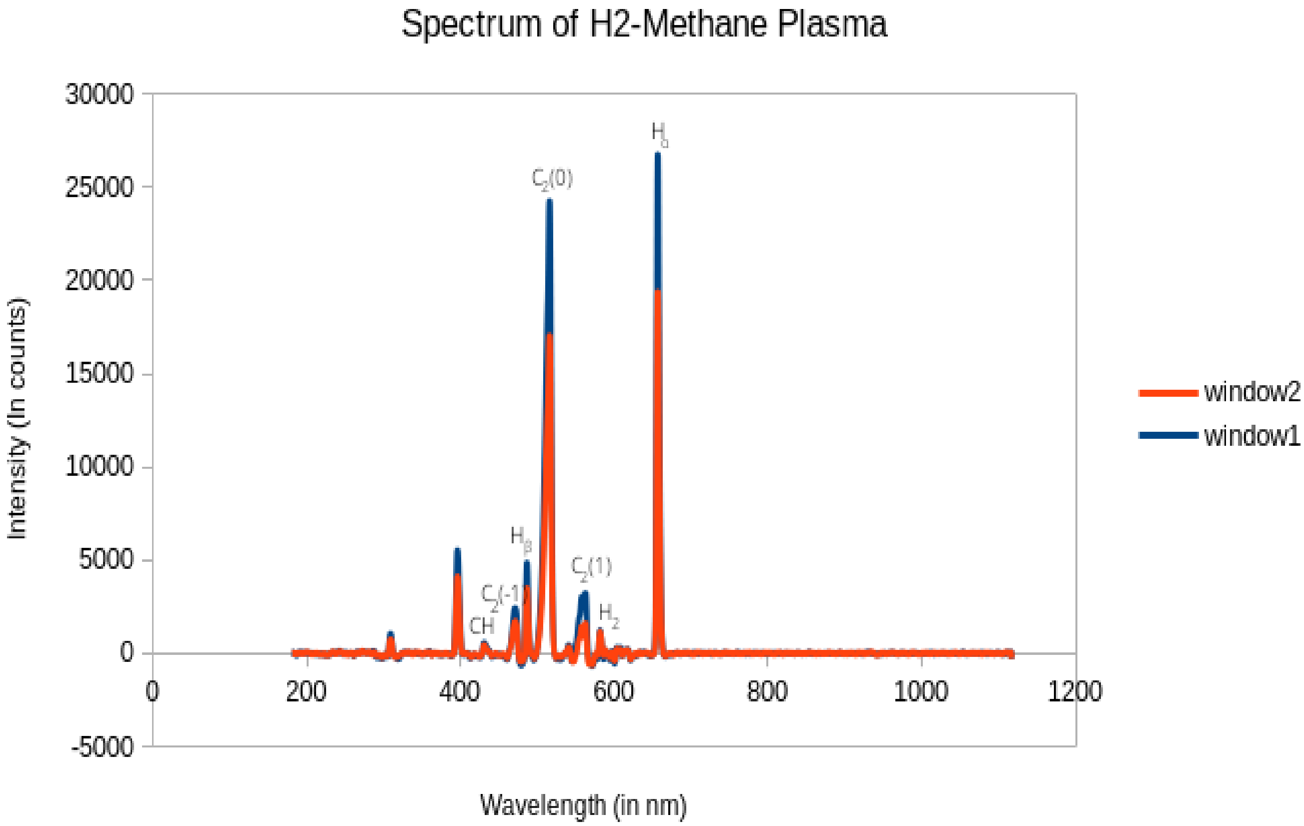
We obtain the electron temperature to be approximately **0.813±0.011 eV**

Results:

The spectrum is identified as below. Labels are marked using GIMP.



Identification of H_α and H_β lines in H_2 plasma.(Wavelength in nanometres)



Identification of H_α,H_β, C_2 swan lines (ν values denoted in bracket), CH and H_2 Fulcher band in $H_2 - CH_4$ plasma.

The electron temperature of the H_2 plasma is 0.813±0.011 eV or 9431.11 ± 133.90 K