

# Construction of Plasma Enhanced Chemical Vapor Deposition Technique Via Commercial Microwave Oven

Nicodemus Kure<sup>1,\*</sup>, Nizar Mohd Hamidon<sup>2</sup>, Isaac Hyuk Daniel<sup>1</sup>, Abdullahi Anderson Kassimu<sup>3</sup>, Sunday Habila Sarki<sup>4</sup>

<sup>1</sup>Department of Physics, Faculty of Science, Kaduna State University, Kaduna, Nigeria

<sup>2</sup>Institute of Advanced Technology, Universiti Putra Malaysia, Selangor, Malaysia

<sup>3</sup>Airforce Research and Development Centre, Kaduna, Nigeria

<sup>4</sup>Department of Science Laboratory Technology, Nuhu Bamali Polytechnic, Kaduna, Nigeria

## Email address

[nicodemus.kure@kasu.edu.ng](mailto:nicodemus.kure@kasu.edu.ng) (N. Kure)

\*Corresponding author

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**Abstract:** In this study, Commercial microwave oven with operating power of 600 W was used to irradiate the vacuum tube at atmospheric pressure of 0.8 mbar with 2.45 GHz frequency, which leads to the formation of plasma in the tubular reactor. The generated plasma utilizes Microwave heating as an alternative to conventional heating. Microwave plasmas are basically electrodeless gas discharge plasma which consists of high frequency electromagnetic radiation in the GHz range. The cylindrical Quartz tube of 30 cm x 10 cm in dimension is inserted horizontally across the microwave cavity. A vacuum pump was used to reduce the pressure of the quartz tube to atmospheric pressure for four (4) minutes. There was a formation of whitish ionize gas when the microwave oven is switch on. The temperature of the plasma ball was measured using K-Thermocouple at 750°C of a ball size of 8 cm x 10 cm in dimension. Pressure, Temperature and Surface geometry are considered as standard parameters for plasma generation.

**Keywords:** Plasma, Microwave Oven, Electromagnetic Radiation, Fluke Multimeter, K-Thermocouple

## 1. Introduction

Commercial microwave oven was invented in 1952, the prevalent usage started few decades after as a result of Japanese technology transfer and global marketing [1]. In general, microwave energy is electromagnetic waves in nature which specifically lie between infrared radiation and radio waves in the region of electromagnetic spectrum with wavelength between  $1 \times 10^{-3}$  m - 1 m, which corresponds to the frequencies that are between 0.3 GHz - 300 GHz. The microwave band have been regulated both national and international levels with operating frequency of 2.45 GHz to minimize interference with communications industries [2].

In recent years, researchers developed tremendous interest

in microwave heating because of its promising future in material synthesis, due to its unique energy transfer efficiency with the use of certain high frequency electromagnetic radiations which gives it advantage over the conventional heating [3]. Materials which produce heat when being irradiated with microwaves are referred to as microwave absorbers.

Plasma is considered as an ionized gas system; consist of electrons, ions and neutral particles. The scientific studied of ionized gas by two American Nobel laureate Langmuir and Tonks, in 1923 give birth to what is known as plasma [4]. A gas is made conductive by providing a high electromagnetic radiation into the gas system, continuous absorption of this radiation leads to plasma formation. Plasma is also called the fourth state of matter. Artificial plasma is generally produced

by application of electric or magnetic fields over certain amount of gas in a confined region [4, 5, 6].

Interaction of dielectric materials with microwaves leads to what is generally described as dielectric heating. The behavior of materials when irradiated in microwave field is described by its dielectric loss tangent:

$$\tan\delta = \varepsilon''/\varepsilon' \quad (1)$$

The dielectric loss tangent is primarily made-up of two important parameters, the dielectric constant (or real permittivity),  $\varepsilon'$ , and the dielectric loss factor (or imaginary permittivity),  $\varepsilon''$  i.e.

$$\varepsilon = \varepsilon' - i\varepsilon'' \quad (2)$$

Where,  $\varepsilon$  is the complex permittivity. The amount of incident energy reflected and absorbed is determined by dielectric constant ( $\varepsilon'$ ), while dissipation of absorbed microwave energy in form of heat is measured by dielectric loss factor ( $\varepsilon''$ ) [2, 7, 8]. Thus, materials with high loss factor are easily heated by microwave energy [9], and the general methods for energy dissipation in materials are mainly due to ionic conduction and dipolar rotation [10].

Furthermore, microwave heating has been proposed by researchers to be an alternative to conventional heating due to its ability to accelerate rate of reaction, its volumetric heating, rapid heating, economical, and clean techniques. Microwave heating basically offers an advantage of having higher heating rate compare to conventional heating. This heating phenomenon deals with the transfer of electromagnetic energy to thermal energy due to molecular interaction with the electromagnetic field. Therefore, the maximum temperature is determined using the dielectric properties of the receptor (material that has ability to absorb microwave energy), and it is believed that materials are heated differently by microwave. This study proposed an alternative technique for heating via commercial microwave oven.

The necessary control parameters that were considered in this study are pressure and temperature. The pressure is used to monitor the vacuum level of the chamber. Lower pressure serve a vital role by allowing the electrons to maintain their energy (temperature) due to elastic collisions [11]. Furthermore, low pressure increases the rate of ionization, which subsequently helps in maintaining plasma stability.

Microwave heating offers an advantage of volumetric heating compared to conventional heating. And the heating phenomenon was due to molecular interaction with the electromagnetic field that results to transfer of microwave energy to thermal energy. However, it is believed that different materials are heated differently by microwaves [12]. The temperature measurement is directly proportional to the time; as temperature increases, the time increases.

## 2. Materials and Method

The experiments were carried-out in tubular reactor

(quartz tube) of length  $55.4 \pm 0.1$  cm, with outer and inner diameter of  $7.1 \pm 0.1$  cm and  $6.6 \pm 0.1$  cm, respectively. The domestic microwave (Samsung M539 MAN200405W) was modified to insert a quartz tube of volume 1860 ml. It consist of a unit of high power regulatory microwave oven, a vacuum pump, Pirani gauge with controlled unit, quartz tube and a teflon cap. Quartz was recommended as safer process instead of glass, plasma may damage the glass. The tube was made vacuum continually throughout the experiment by using rotary vacuum pump (Boc Edwards XDS 5c). Reducing the tube pressure enhances plasma formation. The pressure inside the tube was kept below 1 mbar via Pirani pressure gauge (Boc Edwards APG-M-NW25 ST/ST) and temperature measurement with K-Type Thermocouple (Control thermosb Company). The schematic representation of the experimental setup is shown in Figure 1. Moreover the designed do not require sophisticated process chamber and expensive materials as compare to conventional setup [13].

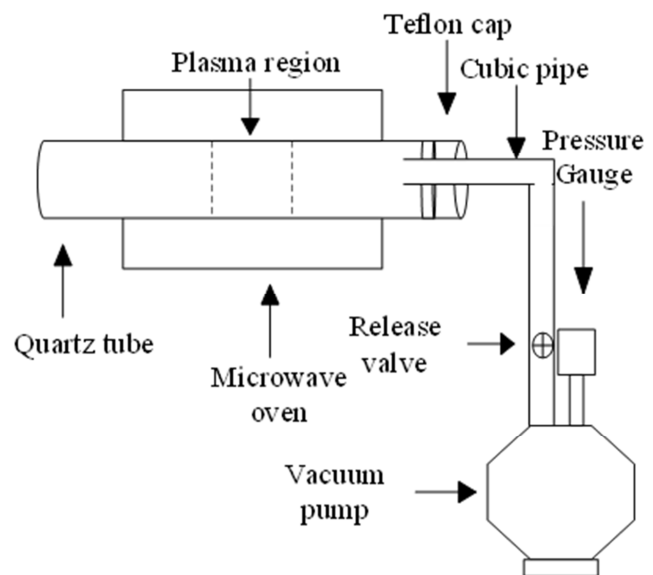


Figure 1. Schematic of experiments setup.

The studies highlight the significant role the parameters play during the plasma generation process. Temperature as one of the parameter selected using K-type thermocouple to investigate the temperature profiling inside the tubular reactor which was done, to establish the temperature measurement of the process. Pressure plays a vital role for plasma generation. With the help of vacuum gauge and pump, the pressure needed for plasma generation was examined. The tube was then irradiated with microwave energy, which ionizes the residual gas inside and continuous absorption leads to plasma formation. The plasma color changes with time due to continuous ionization of the gases within the tube. Low pressure helps in reducing inelastic collision in molecular phenomena, thus electrons maintained their energy (temperature) leading to plasma stability. The temperature reading was recorded using K-type thermocouple sensor until the process was completed. The

modified commercial microwave oven was switched on to initiate the reaction process.

### 3. Results

The plasma starts few seconds after switching on the microwave oven and a bright purple color was observed. Temperature measurement showed the plasma region was 210°C at 30 seconds, then after between 120-240 seconds it remains in the range of 750-820°C. Pressure of 0.8 mbar was maintained.

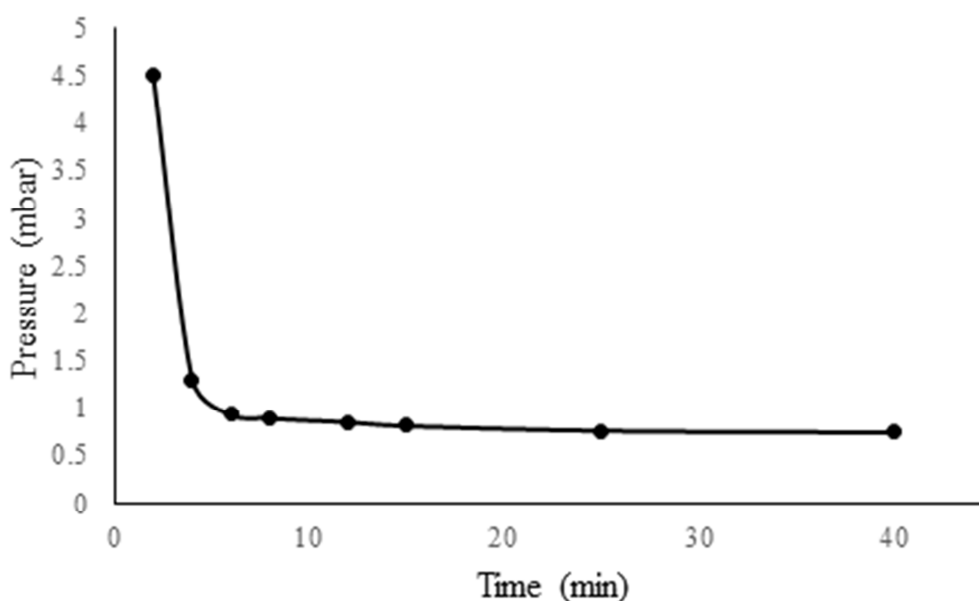


Figure 2. Relationship between pressures against time for plasma generation.

### 4.2. Temperature

The temperature measurement tends to be directly proportional to the time [14, 13]. The temperature measurements was maintained at  $750 \pm 1^\circ\text{C}$  as depicted in Figure 3 [15, 16].

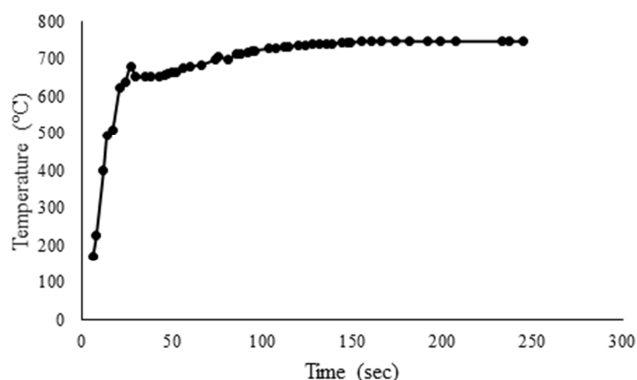


Figure 3. Relationship between temperature and time for synthesis.

### 5. Conclusion

An alternative technique of generating plasma was

## 4. Discussion

### 4.1. Pressure

The vacuum chamber (quartz tube) Pressure measurement was achieved using Active Pirani gauge. The pressure measurement range in this study that enables the formation of plasma is shown in Figure 2. Much time is needed to lower the pressure of the vacuum system. From Figure 2, five (5) minutes period was used to attain  $4.5 \pm 0.1$  mbar, the pressure decrease with increase in time and it remain linear due to equipment (vacuum pump) limitation. Pressure of 0.8 mbar at 25 minutes was maintained.

developed via commercial microwave oven. The pressure and temperature were controlled at 0.81 mbar and  $750^\circ$  respectively. Dimension of the plasma ball is 2 cm x 4 cm. Pressure, Temperature and Surface geometry are considered as standard parameters for plasma generation.

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