

## Article

# The assessment of carbon dioxide dissociation using single mode microwave plasma generator

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**Abstract:** The paper focuses on the interaction between tungsten (W) wire and microwave field in carbon dioxide (CO<sub>2</sub>) atmosphere. Our experimental set-up uses a microwave plasma generator in order to generate the plasma from the metallic wires. The microwave plasma generator contains a cylindrical cavity - TM<sub>011</sub> propagation modes, commercial source (magnetron) having the 2.45 GHz frequency at 800 W microwave and power supply. In the focal point of the cylindrical cavity we have a high energy of the electromagnetic radiation. The metallic wires from this area will be vaporized and ionized having as effect CO<sub>2</sub> dissociation. The electron temperature regarding metallic plasma produced was estimated using the ratio of atomic emission lines acquired by a high definition of the optical multichannel spectrometer. Determination of the CO<sub>2</sub> dissociation quantity was estimated measuring of the carbon monoxide (CO) resulting of the CO<sub>2</sub> dissociation process. We obtained a high electron temperature of the plasma and a strong dissociation of the CO<sub>2</sub>.

**Keywords:** assessment of gases; carbon dioxide dissociation; plasma generator; OES spectroscopy method;

## 1. Introduction

The carbon dioxide (CO<sub>2</sub>) is the most significant long-lived greenhouse gas in Earth's atmosphere. This gas is important because helps to trap heat in our atmosphere, but a gradual increase in CO<sub>2</sub> concentrations in Earth's atmosphere is helping to drive global warming. Many researches concerning CO<sub>2</sub> dissociation used different methods. One of them uses plasma systems to break down CO<sub>2</sub> and release carbon monoxide (CO) and oxygen (O). Plasma is a good environment that allows CO<sub>2</sub> dissociation, because the disintegration process of the gas molecules from plasma is based on the collisions with charged species created in the plasma and neutral particle. Different plasma systems were used in experimental researches such as: dielectric barrier discharges (DBD) [1, 2], inductively coupled radiofrequency plasma [3, 4], microwave discharges [3, 5, 6], glow discharges [7, 8] or gliding arc discharges [9]. The conversion rates varies between plasma systems types, from less than 4% for DBD [10], to less than 35% for microwave discharge and gliding arc discharges [6, 10]. The experimental studies showed that the conversion rate efficiency of the CO<sub>2</sub> is depends of the plasma temperature. The CO<sub>2</sub> molecule begins to split into CO and O<sub>2</sub> near 2000 K. The decomposition of CO<sub>2</sub> can only be carried out at an extraordinarily high temperature (3000–3500 K) [11]. New methods with higher conversion rate efficiency are required.

Therefore our research focused on CO<sub>2</sub> high conversion rate efficiency. To our best knowledge, it is the first time when CO<sub>2</sub> dissociation process was done using plasma resulted from interaction between metallic wires and microwaves. Optical emission spectroscopy method (OES) was used for the electron temperature determination in plasma.

Microwaves are defined as electromagnetic radiation with frequency range between 300 MHz and 300 GHz corresponding to wavelength from 1mm to 1m [12, 13]. Microwaves are non-ionizing radiations with energy between 10<sup>-3</sup> eV and 10<sup>-6</sup> eV/photon.

Dielectric materials can absorb volumetrically the electromagnetic energy from microwave and transform it into heat [14]. Researches concerning microwave applications for metal heating were seldom because it is known that bulk metals reflect microwave radiation and thus cannot be directly heated [15]. Microwave interaction with metals is restricted to its surface only.

Research studies from 2001 have revealed that metallic powders (tungsten carbide- cobalt) that are introduced in a multimode cavity in a microwave field can absorb the microwave radiation [16]. Other studies from in 2009 [17] highlighted that microwave absorption depends on the dimension of the metallic particle and the frequency of the electromagnetic radiation. In 2009 A. Mondal et. al has exposed Cu powders (particles between 6 µm-385 µm) in microwaves in a multimode cavity for 10 min. The experimental results showed that the Cu powder with particle dimension of 6µm reaches temperatures of about 1000° C and reveal that microwave absorption of the metallic powders depends on the depth of penetration [17].

Other recent studies showed that the microwave absorption process can be present in the interaction of non-ionizing radiation with metallic objects that have dimensions much larger than the depth of penetration [18].

In 2010 we reported the microwave absorption by metal wires [18] and we demonstrated that lead metal wires having diameters less than 0.5 mm can be vaporized and ionized by microwaves from the single-mode cylindrical cavity. We used a 800W microwave power and 2.45 GHz frequency. Later, we investigated the possibility of controlling the quantity of metal (lead wires) which is vaporized and ionized in this manner [19].

In 2010 we investigated [20] the microwave heating behaviours of metal wires (lead) in vacuum conditions and we found that at 10<sup>-5</sup> milibar pressure the vaporization and ionization process of the metal wires does not occur and the metal wire is only heated up to melting temperature. The metallic wires can be heated up to plasma temperature in air conditions at a normal atmosphere and at room temperature. In vacuum conditions the metal wires do not exceed the melting temperature. It is possible that air molecules could contribute to the metal microwave absorption process.

In order to investigate if the gas molecules have any contribution to the microwave heating process of metallic wires we used a small Teflon piece. When the metallic wire together with Teflon piece in vacuum conditions was exposed in the microwave field, the metallic wire generated a strong electric field, then Teflon was heated and it was decomposed in gases [21]. This electric field ionized the Teflon gases and the heat generated by the Teflon ion gas could vaporize the metal wire. Then the metal vapours were ionized in the microwave electric field from cylindrical cavity.

Other similar experiments were made in 2015 were it was used a rectangular cavity to create titanium plasma from titanium samples [22] and copper plasma from a copper sample [23]. The plasma column was created when an electrode (titanium or copper) is brought into contact with a plate while irradiated by microwaves at 2.45 GHz generated by a 1-kW magnetron.

The objectives of present research are to investigate the dissociation of the carbon dioxide (CO<sub>2</sub>)

following the absorption processes of the microwave radiation by tungsten (W) wire using a microwave plasma generator in order to generate the plasma from a noncontact tungsten electrode.

The optical emission spectra of the metallic plasma were obtained in the region of 200–850 nm and have been recorded by Ocean Optics USB 2000+ spectrometer. Plasma was created at 700W microwave power in a CO<sub>2</sub> atmosphere, at normal pressure and ambient temperature. The CO quantity from CO<sub>2</sub> atmosphere was analysed using the CHEMIST 504S gas analyser kit instrument.

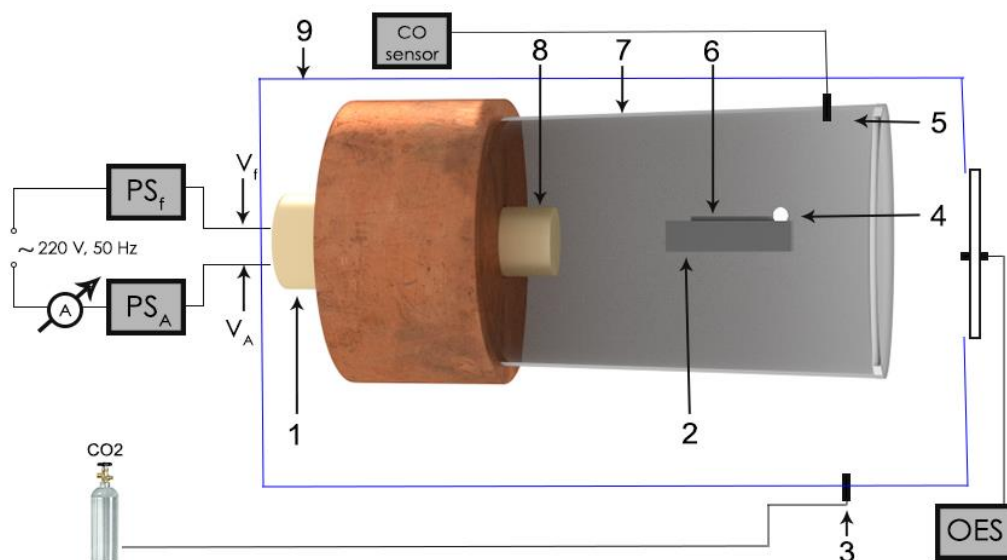
## 2. Materials and method

Dissociation of the CO<sub>2</sub> molecules using the microwave field is an interesting area because from the scientific point of view the microwaves are non-ionization radiation, but in the same time the microwave photons does not have enough energy for the ionization of the CO<sub>2</sub> molecules.

According to the formula (1) [9] the endothermic plasma-chemical process of carbon dioxide decomposition is:  $\text{CO}_2 \rightarrow \text{CO} + 1/2\text{O}_2$ ,  $\Delta H = 2.9 \text{ eV/molecule}$ , (1)

The energy required to start the process of total decomposition of the CO<sub>2</sub> follows the formula (2) [9].  $\text{CO}_2 \rightarrow \text{CO} + \text{O}$ ,  $\Delta H = 5.5\text{eV/molecule}$  (2)

Therefore, in our experiment the dissociation of the CO<sub>2</sub> molecules is based on the interaction between microwave fields with metallic wire in a CO<sub>2</sub> atmosphere (Fig.1).



**Figure 1.** Experimental design of the CO<sub>2</sub> dissociation:

1 – magnetron, 2- ceramic support, 3- connector CO<sub>2</sub>, 4- high electromagnetic energy region, 5- CO<sub>2</sub> sensor, 6- metallic wire, 7- cylindrical cavity, 8- magnetron antenna, 9 – pressure chamber.

In the focal point of the cylindrical cavity, where is placed the metallic electrode is generated a high electrical field (from 3 to 6 MV/m [24]). The metallic wire (W) from this area will be vaporized and the CO<sub>2</sub> molecules will be break down having as a final result oxygen and other molecules.

The experimental device used for the CO<sub>2</sub> dissociation was a microwave plasma generator presented in figure 2.

The microwave plasma generator consists of a cylindrical cavity, a microwave source (commercial magnetron) with  $\nu=2,45 \text{ GHz}$ , 800W emitted power and a power supply (one high

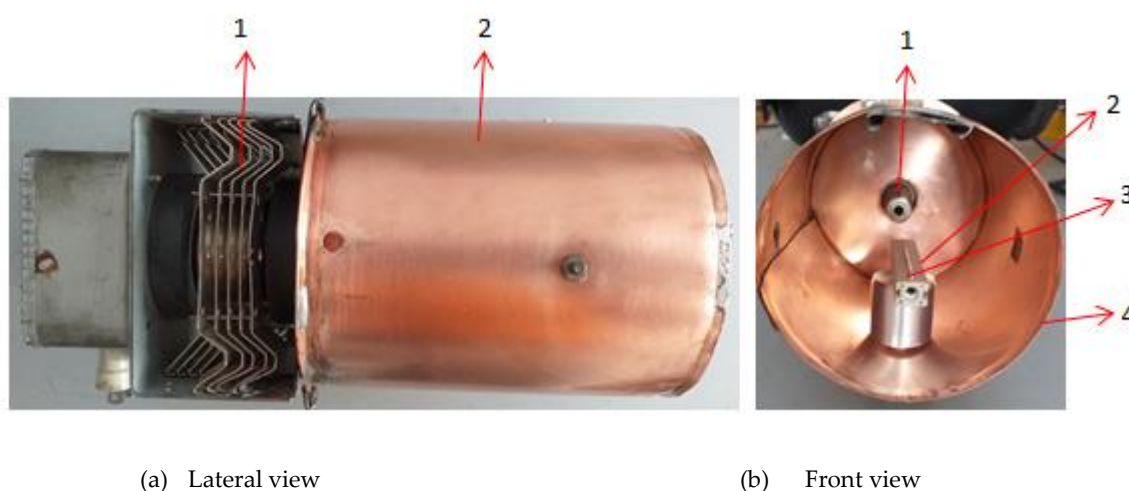
voltage power supply for the magnetron anode - PSA and one low voltage power supply for the filament of the thermo-electronic source of the magnetron - PSF).

The PSA have an electronic module that can help to modify the length of the electric impulses (duty factor) of the source that feeds the anode magnetron. Therefore, by changing the duty factor of the anode voltage from the magnetron, we modify the quantity of the vaporized metal and as final results we obtain different quantity of the dissociated CO<sub>2</sub>.

Cylindrical cavity:

The  $TM$  cylindrical cavity dimensions were calculated with the following formula (3) [25, 26].

$$(f_r)^{TM}_{mnl} = \left( \frac{1}{2\pi\sqrt{\mu\epsilon}} \right) \cdot \sqrt{\left( \frac{p_{01}}{a} \right)^2 + \left( \frac{l\pi}{h} \right)^2} \quad (3)$$



**Figure 2.** The microwave plasma generator:

(a) 1 – magnetron, 2- cylindrical cavity; (b) 1 –magnetron antenna, 2 – ceramic support, 3 –metallic wire

Where:

$a$  - radius of the cylindrical cavity (m);  $h$  - height of the cylindrical cavity (m);

$l$  - Longitudinal mode of the cavity;

$\mu$  - Permeability of the medium within cavity (H/m);

$\epsilon$  - Permittivity of the medium within the cavity (F/m);

$p_{01}$  - First zero of the Bessel function (equal to approx. 2.405);

$f_r$  - The resonant frequency of the cavity.

The indices  $mnl$  of the  $TM$  propagation mode refers to the number of half-wavelength variations in the radial, axial and longitudinal directions. The optimal dimensions of the  $TM_{011}$  cylindrical cavity in our experimental data are the following: diameter of the cavity is 11 cm and the length is 10.5 cm. The metallic wire used has 0.5 mm diameter with 50 mm length. The sample were placed on a ceramic support and introduced in the cavity (Fig.2). The high-density energy region (focal point) is located at 6.1 cm distance from the magnetron antenna, which corresponds to half wavelength for 2.45 GHz microwave frequency.

According to the Poynting equation (4) [27] when the microwave power in the cylindrical cavity increases, the electric field in the metallic wire increases as well. The microwaves induce strong

electric fields in metallic wires and this electric field produce the ionization of the gas surrounding metallic wire from high-intensity area. The ions of the plasma interact with metallic wire and vaporize the metallic wire.

$$\vec{P} = \frac{1}{2} \text{Re}(\vec{E} \times \vec{H}^*) = \frac{1}{2} \frac{|E|^2}{\eta} \quad (4)$$

Where,  $P$  = Poyting vector,  $E$  = electric field,  $H$  = magnetic field,  $\eta$  = impedance of medium

The relationship between the microwave electric field and the current density (conduction and displacement current) from metallic wire is:

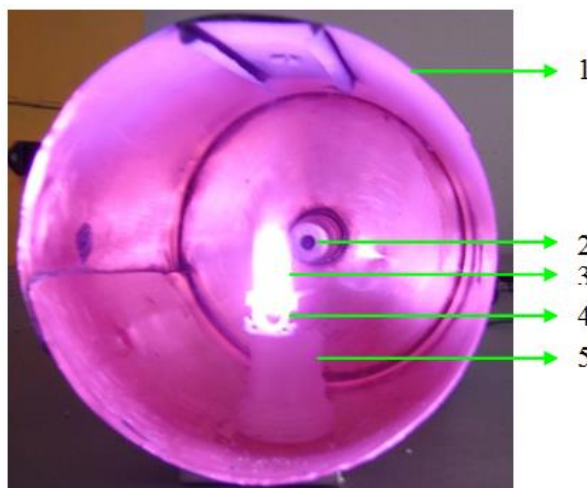
$$J = (\sigma + j\omega\epsilon)\vec{E} \quad (5) [26];$$

Where:  $J$  = current density in metallic wires,  $\sigma$  = electrical conductivity of metallic wires,

$\omega$  = angular frequency,  $\epsilon$  = electrical permittivity,  $E$  = electric field

The metallic particles are ionized in the microwave field because these metallic particles have similar or smaller dimensions than the skin depth of the microwaves.

The vaporization rate of the metallic wire depends on the electrical conductivity of the metals at a certain temperature [28]. Therefore, the dissociation of the  $\text{CO}_2$  molecules is produced through interaction between electric charges induced by microwaves in noncontact metallic electrode with  $\text{CO}_2$  molecules and interaction between ions of the metal with  $\text{CO}_2$  molecules. In our microwave  $\text{CO}_2$  dissociation experiment we chose tungsten (W) as a noncontact electrode because this metal has good electrical conductivity which leads to a small rate of metal vaporization (Table 1). The tungsten wire is slowly consummated during the  $\text{CO}_2$  dissociation process and therefore we can dissociate  $\text{CO}_2$  for a long time (Fig.3).



**Figure 3.** The metallic wire exposed in the microwave field: 1- cylindrical cavity, 2- magnetron antenna  
3- metallic plasma, 4- ceramic support, 5 -plastic support.

**Table1.** Energetic parameters and vaporization rate of the tungsten

Tungsten wire	First ionization energy (eV)	Vaporization rate (mg/s)	Electrical conductivity (S/m)
(0.5 mm diameter)	7,98	2	$2 \cdot 10^7$

First step in our experiment was to introduce the W wire inside the cylindrical cavity (Fig.2). The pressure chamber was filled with  $\text{CO}_2$  at atmospheric pressure (Fig.1).

Because in the  $TM_{011}$  propagating mode, the microwave electric field propagates along the axis of the cavity, the metallic electrode must be positioned along and as close as possible to that axis.

One end of the metallic electrode was placed in the focal point of the cylindrical cavity and after the interaction between the microwave and the metallic electrode, we obtained: metallic particles, CO and O (Figs. 4, 5). The plasma characterization has been determined by the optical emissions spectroscopy method (OES) [29, 30]

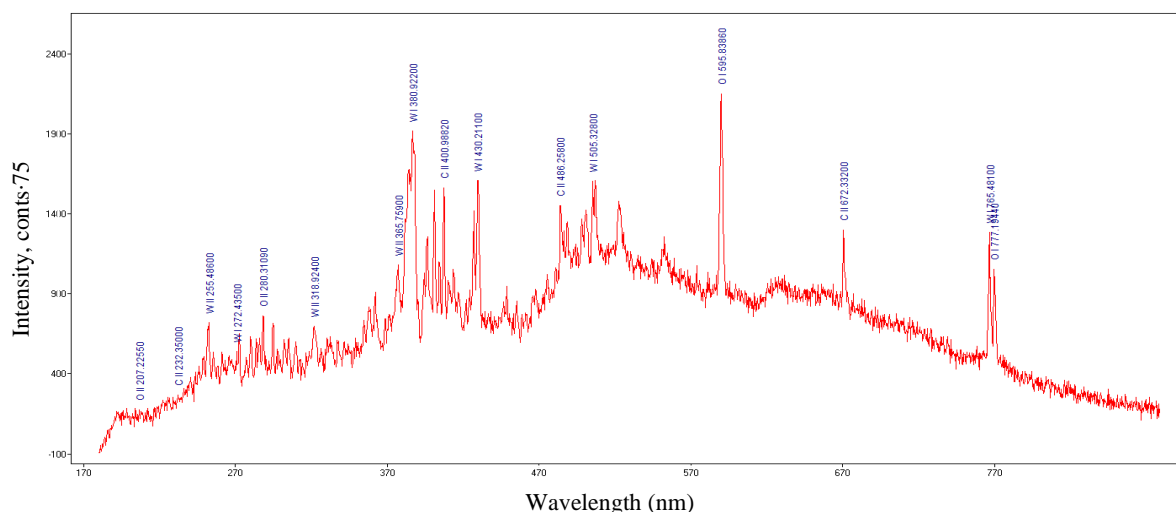
The optical emission spectra of the metallic plasma were obtained in the region of 200–850 nm and have been recorded by Ocean Optics USB 2000+ spectrometer. Plasma was created at 700W microwave power in a CO<sub>2</sub> atmosphere, at normal pressure and ambient temperature. The CO quantity from CO<sub>2</sub> atmosphere was analysed using the CHEMIST 504S gas analyser kit instrument.

### 3. Results and discussion

Microwave plasma is widely investigated in view of their potential to provide highly efficient CO<sub>2</sub> conversion. The high efficiencies have been explained by vibrational excitation that had as result a strong non-equilibrium between vibrational and translational degrees of freedom, which in turn intensify dissociation [31].

In our experiment, the dissociation of the CO<sub>2</sub> is based on the microwave absorption process by metals with the process characterized only by Ohm losses [17]. The metallic wire was exposed for 20 seconds at 700W microwave field in CO<sub>2</sub> at normal atmospheric pressure. During the vaporization process of the metallic wire in a cylindrical cavity was generated metallic vapors and other gases such: CO, O<sub>2</sub>, O, C, etc. (Figs. 4, 5). The dissociation of the CO<sub>2</sub> was investigated through the plasma characterization technique and determination of the carbon monoxide (CO) resulting from the CO<sub>2</sub> dissociation process. The optical emissions spectroscopy method was used for plasma characterization and electron temperature determination.

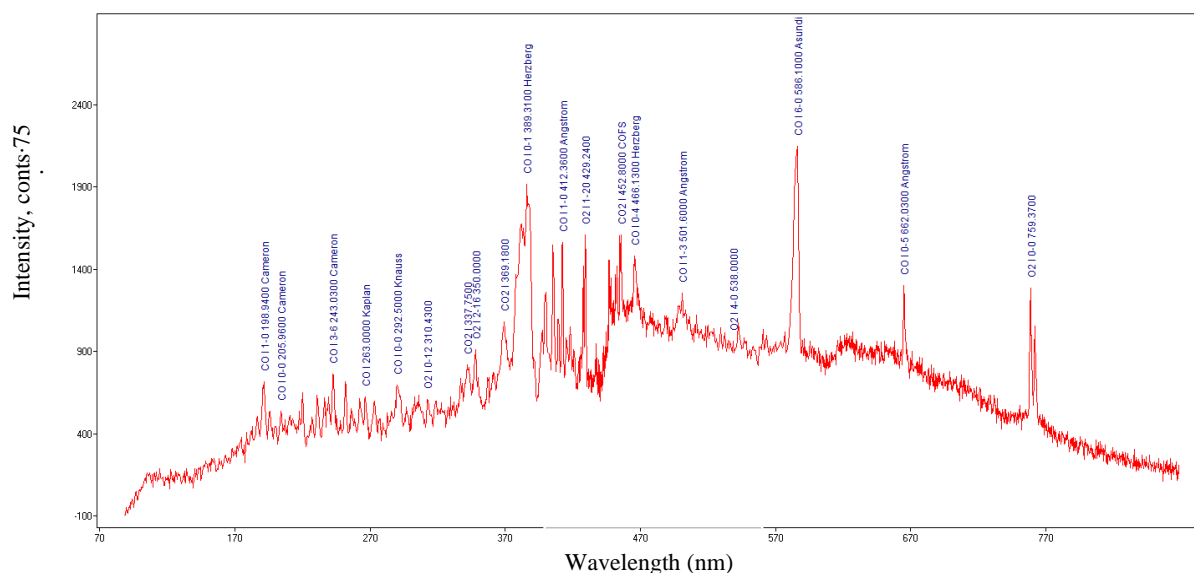
The volume of the pressure chamber using in this experiment was 0,024m<sup>3</sup> (24 liters).



**Figure 4.** Optical emission spectra of the plasma: Ions generating during CO<sub>2</sub> dissociation process.



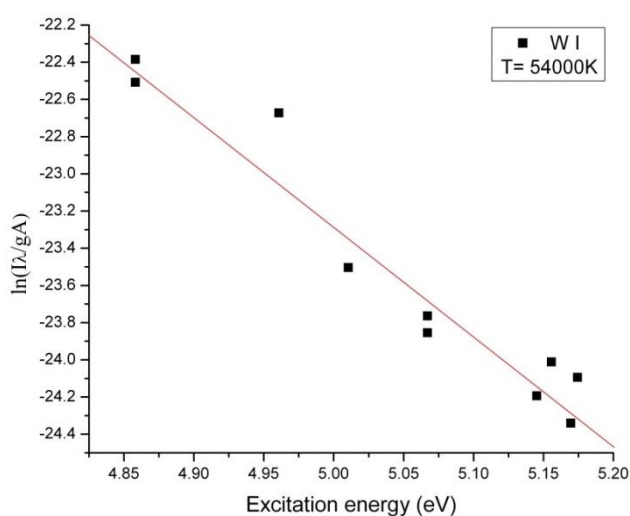
Spectrum Analyzer software [32] was used for optical emission spectra plasma identification. The microwave field creates ions of metal, oxygen, and carbon. During the  $\text{CO}_2$  dissociation process other gases molecules are formed [33, 34]. These reactions are observed from the emission spectra where it shows the main plasma components: oxygen (OI and OII), carbon (CII) and tungsten (WI and WII) (Fig. 4). Because in focal point of the cavity is generated high electromagnetic energy in plasma was created two-stage ionization ions: OII = 35.117eV, CII = 24.382eV, and WII = 17.619 eV.



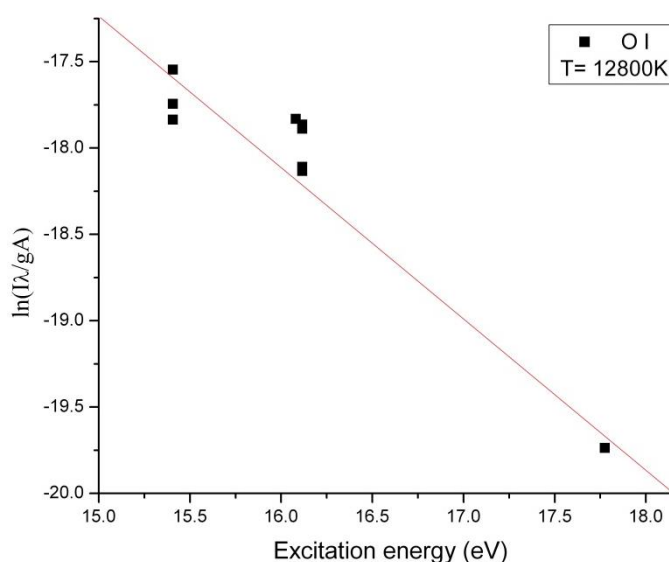
**Figure 5.** Optical emission spectra of the plasma: The monoxide and oxygen generated during the  $\text{CO}_2$  dissociation process

The monoxide and oxygen is generated during the  $\text{CO}_2$  dissociation process (Fig.5). During the recording of the emission, spectrums were used 1ms integration time. The electron temperature of tungsten plasma and oxygen plasma was determinate using the Boltzmann plot method, which assumes that local thermodynamic equilibrium-LTE is met within the plasma [21].

The electronic temperature for tungsten ions is 54000 K (Fig.6), and the electronic temperature of the oxygen ions is 12800K (Fig. 7).



**Figure 6.** The Boltzmann plot and electron temperature for ions tungsten



**Figure 7.** The Boltzmann plot and electron temperature for ions oxygen

In pressure chamber the concentration of the CO in the CO<sub>2</sub> atmosphere was 3ppm. After the 20 seconds reaction time, (interaction time between microwave field and metallic wire), the quantity of the CO generated during the CO<sub>2</sub> dissociation process was increased to 1377ppm.

The CO quantity was measured using the CHEMIST 504S gas analyzer kit instrument. The instrument uses the electrochemical sensor to measure concentration of CO. The resolution of the instrument for CO is  $2 \times 10^4$ ppm.

#### 4. Conclusions

We have highlighted the CO<sub>2</sub> was dissociated through interaction between metallic wires and the microwave field. Tungsten wire with 50 mm length and 0.5 mm diameter was used as a non-contact electrode and the electrode was exposed to the 700W microwave power for 20 seconds in a CO<sub>2</sub> atmosphere. During the interaction with the microwave field, this was vaporized and ionized and the energy to create plasma comes from the microwave field inside the cavity. Because the focal point of the cavity generates a high electromagnetic energy, were created two ionization stages in plasma. The electron temperature of the plasma was 54000K for WI and 12800K for OI.

After the interaction between the microwaves and the non-contact electrode, we obtained tungsten particles, CO and O. After 20 seconds reaction time the quantity of the CO increased from 3 ppm up to 1377ppm inside in the pressure chamber.

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**Conflicts of Interest:** The authors declare no conflict of interest regarding the publication of this paper.



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