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Towards a miniature wobbe index meter

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Summary

The aims for this thesis is to investigate the possibility of making stable combustion of methane in the air on a small scale, and the possibility of creating a small-scale combustion device with 3D printing technology of aluminum oxide.

In general, realizing stable combustion in small scale is never easy as doing so in macro scale. Basically, two problems can happen in small-scale combustion. One is called 'flame quenching,' the 'flame quenching' is a phenomenon that the flame on a small scale would be extinguished because of some unique physical characteristics of the small combustion chamber. Another one is the materials of the small combustion chamber are not able to withstand the high power generated by flame. Therefore, in this thesis, three different combustion systems are designed to overcome these problems and realize a stable flame in different small scale. The first design is to realize a mini scale combustion in a small quartz tube. The second one is to realize microscale combustion with a fabricated tiny chip. The third one is based on the first design, but instead of realizing mini scale combustion on quartz tube, microscale combustion is going to be generated inside a trumpet shape aluminum oxide tube.

Because the reason of timing and material preparing, the second design has been finally realized, and three experiments have been done subsequently to verify if the combustion could happen in such a tiny chip. At first, there was almost no sign of combustion. After some adjustment was made, there were some indications of temperature rise in the combustion chamber, it was suspected as combustion.

On the other hand, a 3D draft for the third design was made, and this draft had been sent to a 3D printing company to see if it's possible to create such a tube with aluminum oxide.

In short, 'likely' combustion is indicated by the temperature data of the experiments, but future experiments are needed to verify if it is real combustion. Also the feasibility of 3D printing the combustion chamber is needed to be confirmed until the reply from this company is given.

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Introduction

Natural gas serves as an essential energy source in our daily life. Regarding the fact that the Dutch gas quality bands are expected to vary a lot because the main gas supply stream in The Netherlands changes in upcoming years. Therefore, it is critical to determine the value to pay per mega-Joule of heat generated per cubic meter of gas ($\text{euro}/\text{mJ}/\text{m}^3$). The traditional measurement devices may lead to some problems such as large space occupation, low portability, and over-consumption of burning gas. For the sake of optimizing the measurement devices, the possibility for miniaturizing the gas burning meter and realize it with a fabricated chip is discussed. However, the realization of steady and continuous combustion in microscale is quite different from in macro scale. Some problems like flame quenching which will extinguish fire may happen in small space burning system. On the other hand, if the combustion power is beyond the limitation of which chip material can withstand, the chip may break down as well. Therefore this assignment mainly focuses on investigating:

- The feasibility for creating a steady burning fire on the small scale.
- The possibility of 3D printing a combustion chamber with high melting point material—aluminum oxide.

Theory

This chapter mainly gives an introduction to the concept of the Wobbe index, the general structure and the fundamental working principle of the Wobbe meter. Also some technical features and requirements for each part of the Wobbe index meter are introduced in this chapter.

2.1 Wobbe index

Wobbe index, proposed by Italian engineer Goffredo Wobbe in 1927, serves as an indication of heating values of the burning gas. It can be used to compare the energy released by burning gases at standard pressure and temperature. For instance, if two kinds of gas have the same Wobbe index, the burning heat value of those gases should be the same at the same pressure. The definition of Wobbe index is expressed as follows:

$$WI = \frac{H}{\sqrt{GS}} = \frac{H}{\sqrt{\frac{\rho_{\text{gas}}}{\rho_{\text{air}}}}} \quad (2.1)$$

[1]

Where H is the calorific value of the burning fuel with unit $[J/m^3]$, the GS is the specific gravity of the fuel respect to air which also equals the density of the burning gas divided by the density of the air. The calorific value will be decided by the measurement value of increasing temperature due to burning divided by the heat capacity of the temperature sensor. The GS depends on which kind of gas is going to be measured plus the temperature and pressure of the environment during the experiment.

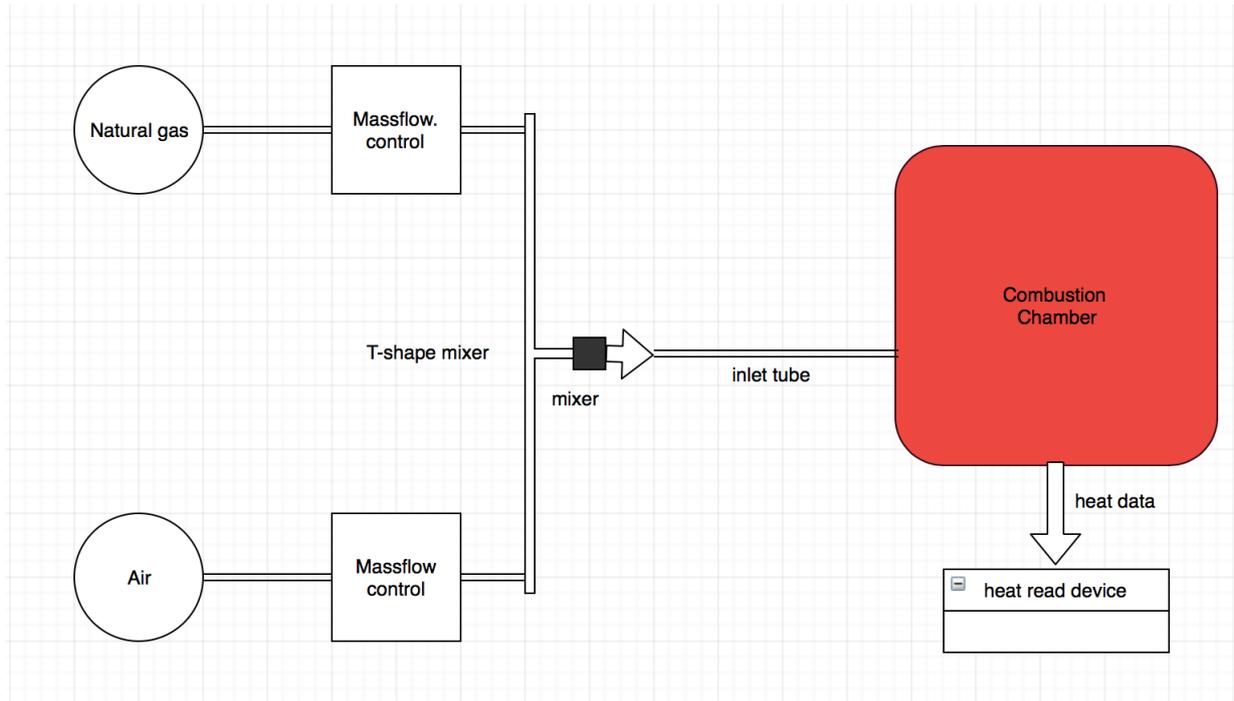


Figure 2.1: General schematic of Wobbe index meter

Components of Wobbe index meter
Gas tanks
Mass flow controller
Mixer
Combustion chamber
Heat read device

Table 2.1: Components of Wobbe index meter

2.2 Wobbe index meter structure

The devices used to measure the burning heat value which is introduced in section 2.1 is called 'Wobbe index meter'. Generally speaking, the Wobbe index meter consists of six parts. Two gas tanks containing natural gas and air are put separately in the front. The mass flows of two gas tanks are controlled by two mass flow controllers separately. Passing through the mass flow controller, two gases are going to encounter each other at the mixer and form a homogeneous mixture inside the inlet tube. The mixture is going to react inside the combustion chamber, and the reaction heat is going to import to a heat reading device. The structure is shown in figure 2.1, the components of a Wobbe index meter in indicated in table 2.1.

2.3 Technique requirement for mass flow controller

The mass flow controller needs to control the mass flow. Also, the mass flow decides the velocity of the gas mixture and reaction power indirectly, so the values of mass flow are calculated based on the velocity of the gas mixture and reaction power.

2.4 Technique features and requirements on fluid dynamics

Technique features and requirements on fluid dynamics are essential to study the behaviors of gases travel inside the Wobbe index meter. These behaviors directly decide some crucial factors for creating a stable flame. Therefore the technical features and requirements on fluid dynamics are introduced below.

2.4.1 Average flow velocity

The average speed of a gas through a pipe is the average velocity of the fully developed internal laminar flow. The solution of the average velocity can be obtained from the empirical solution for the mass flow rate since the mass flow rate of the internal flow is defined as:

$$\dot{m} = \rho V_{\text{avg}} A_c = \int_{A_c} \rho u(r) dA_c \quad (2.2)$$

[2] where the ρ is the density of the flow, the A_c is the cross-section area of the tube, V_{avg} and $u(r)$ represents the average and local velocities. The average velocity of the laminar flow in a circular pipe with radius R could be derived as:

$$V_{\text{avg}} = \frac{\int_{A_c} \rho u(r) dA_c}{\rho A_c} = \frac{\int_0^R \rho u(r) 2\pi r dr}{\rho \pi R^2} = \frac{2}{R^2} \int_0^R u(r) r dr \quad (2.3)$$

2.4.2 Flow regime inside a pipe

[3] The equation 2.3 is valid when the flow regime is laminar. Generally speaking, there are four kinds of possible flow regimes inside a pipe: Stoke, laminar, transition and turbulent flows. In stoke flow, the inertia force is much less than the viscous force. The laminar flow is the kind of flow with smooth streamlines and highly ordered motion profile. The transition flow is the flow fluctuates between laminar and turbulent flow. The turbulent flow is the kind of flow with highly fluctuated velocity streamline and highly disordered motion profile.

Reynold number for pipe flow	Flow regime
$Re \leq 1$	stoke flow
$1 < Re \leq 2100$	laminar flow
$2100 < Re \leq 4000$	transition flow
$Re > 4000$	turbulent flow

Table 2.2: The relation between Reynold number for pipe flow and flow regime.

The flow regime depends on the ratio between the inertia force of the gas flow and the viscous force generated by the environment. This ratio is also called the Reynold number. The Reynold number for fluid in a pipe expresses as:

$$Re = \frac{V_{avg}D}{v} = \frac{\rho V_{avg}D}{\mu} \quad (2.4)$$

Where V_{avg} is the average velocity of the flow, D is the diameter of the circular pipe, v is the kinematic viscosity, ρ is the density of the fluid, and μ is the dynamic viscosity of the flow.

For non-circular pipe, the Reynold number depends on the hydraulic diameter:

$$D = \frac{4A_c}{P} \quad (2.5)$$

[4]

The hydraulic diameter defines as the ratio between four times of the cross-section area and the cross-section circumference.

The relation between Reynold number for pipe flow and flow regime is shown in table 2.2.

To get a continues steady burning process, in this assignment, the gas mixture flow in the fully developed region of the pipe should be laminar flow.

2.4.3 Gas mixing

For obtaining steady continuous combustion, a homogeneous gas mixture forms inside the inlet tube, this process is related to diffusion. Diffusion stands for a physical phenomenon that the atoms or molecules of two adjacent layer move from high concentration region to low concentration region. In the model of the mixed gas, diffusion is considered as the mass transportation among different gas components, and there should be enough space for gas and air to transport into each other fully, this space is related to the diffusion length [5]:

$$L_d = \sqrt{\alpha D t} \quad (2.6)$$

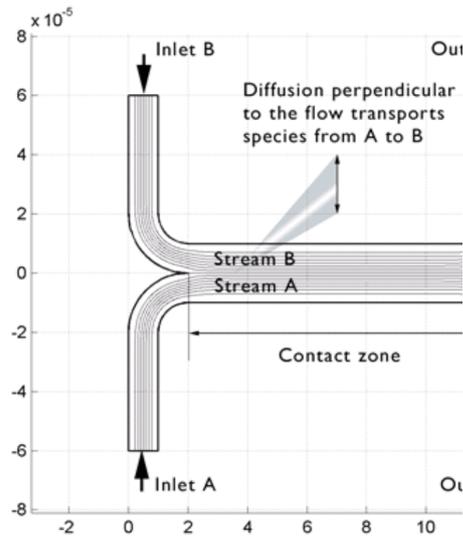


Figure 2.2: Demonstration of gases mixing inside a straight pipe [6]

Where the α is the dimensional factor, D is the diffusivity of the gas, t is the diffusion time. Since in the circular pipe the diffusion times along all diameter directions are all the way the same, in this case, only the two dimensions are interested in. For two dimensional diffusion, the α equals 4.

The diffusion length indirectly decides the maximum inlet tube length for gases getting fully diffused. As shown in figure 2.2, two fluids start to mix and diffuse to each other along the radial direction of the pipe. For the sake of calculating maximum inlet tube length for fully mixed gas, the maximum diffusion time is considered. According to 2.4.3, the diffusion time could be derived as:

$$t = \frac{L_d^2}{4D} \quad (2.7)$$

So the largest diffusion time happens when the diffusion length equals the diameter of the pipe:

$$t_{\max} = \frac{(2R)^2}{4D} = \frac{4R^2}{4D} = \frac{R^2}{D} \quad (2.8)$$

For certain mass flow, according to 2.2, the average flow velocity could be expressed as:

$$V_{\text{avg}} = \frac{\dot{m}}{\rho A_c} = \frac{\dot{m}}{\rho \pi R^2} \quad (2.9)$$

Thus the maximum inlet tube length for obtaining a homogeneous gas mixture is:

$$L_{\max} = t_{\max} * V_{\text{avg}} = \frac{R^2}{D} * \frac{\dot{m}}{\rho \pi R^2} = \frac{\dot{m}}{D \rho \pi} \quad (2.10)$$

2.5 Technique features and requirement for combustion chamber

This section is related to technical features for the combustion chamber to stabilize the flame. Plus, some background information on thermodynamics. The combustion chamber needs to supply enough heat for natural gas to create a stable and continuous fire. The material of the combustion chamber should prevent the radical flame quenching also should be isolated enough to avoid heat transfer to the environment as much as possible.

2.5.1 Temperature of combustion chamber

In this experiment, methane is going to serve as the fuel gas for combustion, so the temperature inside the combustion chamber should be above the ignition point of the methane, and the autoignition temperature of methane is around 537°C [7].

2.5.2 Temperature rise

Studying the temperature rise due to combustion is very essential. It directly decides if the material of the combustion chamber would break down during combustion, also in this experiment, it is the most important indication of combustion. The Temperature rising is calculated as

$$\text{Temperature rising} = \frac{\text{Combustion heat} - \text{Heat transfer to the environment}}{\text{Heat capacity of the combustion chamber}} \quad (2.11)$$

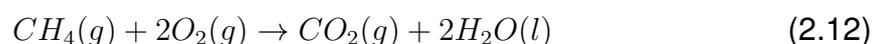
Combustion heat

Generally speaking, the heat of chemical reaction can be calculated as

Heat of reaction = [the sum of all heats of formation of all products] – [the sum of heats of formation of all reactants]

[8]

The balance equation of reaction of methane is represented as:



So the heat generation Δh of methane combustion with sufficient oxygen is calculated as

Heat of reaction = heat of formation of CO₂ (g) + 2 × heat of formation of H₂O (g) – [heat of formation of CH₄ (g) + 2 × heat of formation of O₂ (g)]

For one mole methane the heat generation is

$$[-393.5kJ + 2 \times (-241.8kJ)] - [-74.8kJ + 2 \times (0kJ)] = -802.3kJ \quad (2.13)$$

Heat transfer to the environment

[9] Basically with the help of temperature difference the heat transfers from the combustion chamber to the environment in three different modes, namely conduction, convection, and radiation:

1. When two objects put adjacently, the heat will automatically transfer from high-temperature object to low-temperature object in the form of conduction. For heat conducted through a wall with a thickness Δx and surface area A , the heat conduction is defined as:

$$\dot{Q}_{\text{cond}} = kA \frac{T_1 - T_2}{\Delta x} \quad (2.14)$$

The K is called the thermal conductivity which unit is $W/(m^2 \cdot K)$. The thermal conductivity serves as an important indication of the ability of heat conduction. For heat conduction through a cylinder wall of the pipe:

$$\dot{Q}_{\text{cond}} = \frac{2\pi Lk}{\ln \frac{r_2}{r_1}} \quad (2.15)$$

With L the tube wall length, r_2 and r_1 is the outer and inner radius of the tube.

2. When fluid flows over the surface of one object, it will take some heat out of the object, and this heat transfer mode is called convection. Newton's law of cooling can describe the physical behavior of convection:

$$\dot{Q}_{\text{conv}} = hA_s(T_s - T_\infty) \quad (2.16)$$

Which the h is the convection coefficient and A_s is the surface area where the heat transfer happens, T_s is the temperature of the surface and the T_∞ is the temperature of the fluid which is far away from the surface.

3. Radiation transfers heat energy in the form of electromagnetic waves or photons. The radiation power describes as:

$$\dot{Q}_{\text{rad}} = \delta \epsilon A_s (T_s^4 - T_{\text{surr}}^4) \quad (2.17)$$

With δ is the Stefan-Boltzmann constant, ϵ is the emissivity of the material gives an approximation on how does a surface close to the blackbody, T_s indicates the temperature of the surface, T_{surr} is the temperature of the surrounding.

Thermal resistance

[10] Thermal resistance shows the ability for material to resist the heat transfer, the thermal resistance could be derived from the equations of heat transfer in section 2.5.2:

- For thermal resistance of a straight wall:

$$R_{\text{conduction}} = \frac{\Delta x}{KA} \quad (2.18)$$

- For thermal resistance of a cylinder wall:

$$R_{\text{conduction}} = \frac{r_1 - r_2}{4\pi r_1 r_2 K} \quad (2.19)$$

- For thermal resistance of a convection flow:

$$R_{\text{convection}} = \frac{1}{hA} \quad (2.20)$$

- For thermal resistance derived from radiation:

$$R_{\text{radiation}} = \frac{1}{\delta \epsilon A_s (T_s^2 + T_{\text{surr}}^2)(T_s + T_{\text{surr}})} \quad (2.21)$$

Specific heat capacity

Specific heat capacity is an essential physical quantity in thermodynamics which indicates the temperature change when a certain amount of heat adds to the certain mass of an object. The heat capacity expresses as the ratio of total heat absorbed by the object to the temperature change multiplies the mass of the object :

$$C = \frac{Q}{\Delta T \times m} \quad (2.22)$$

[11]

Where the Q is the net heat absorbed by the object, the ΔT is the temperature change triggered by the heat absorption, and m is the mass of the object, the unit for the Specific heat capacity is $J/(K \cdot kg)$. On the other hand, the when temperature rising after certain amount heat absorbs by the object can be derived as:

$$\Delta T = \frac{Q}{C \times m} \quad (2.23)$$

Flame quenching in the micro combustion

The net effect of conduction, convection, and radiation will lead to a severe problem in combustion chamber called flame quenching. It is because that the heat generation of micro combustion is much less than the heat transfer outside of the combustion chamber. However, as introduced in section 2.5.1, since the temperature of the combustion chamber is above the ignition point of the gas mixture, this problem can be avoided because the gas will always combust in this situation.

Design and calculation

In this assignment small combustion system is going to be designed based on the structure of the Wobbe index meter introduced in 2.2. There are three scenarios of designs for this small combustion system. The components or factors which influence the stabilization of the combustion will be designed and calculated in this chapter.

3.1 Scenario one: Quartz tube burning method

The first scenario is to realize mini-scale combustion in a quartz tube. This quartz tube has an inner diameter $5mm$ and length $30cm$, the end $10cm$ of the tube which is the combustion chamber inserts inside an oven. The rest $20cm$ of the tube stays outside of the oven serves as the inlet tube. One side of the tube is connected to the mixer where the fuel-air mixture enters, at the end of the tube the exhaust gas comes out. The oven serves as the heating source, and a thermocouple is going to measure the increasing temperature as a result of fuel burning. Thermocouple is a kind of widely used temperature sensor, basically the thermocouple is made of two types of unlike electrical conductors which form electrical junctions under different temperature condition. This junction which results the thermoelectric effect will lead to a consequence of voltage difference. This voltage differences can be used as an indicator of temperature [12]. Some important features are calculated. Figure 3.1 demonstrates the schematic of this design, some important components that can directly influence the performance of the flame are indicated in table 3.1.

Basically, the design procedures are:

1. First step, decide the oven temperature for maintaining continuous combustion.
2. Second step, decide the mass flows of air and methane which could make the flame stable inside the tube.

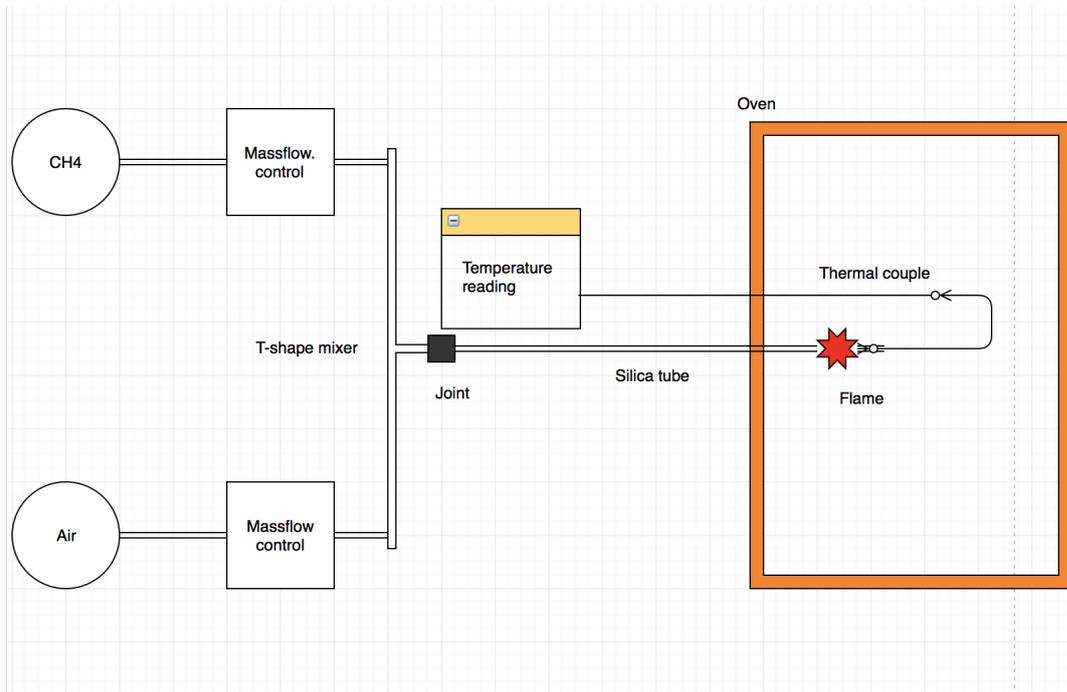


Figure 3.1: Schematic of quartz tube burning method

Inlet tube	First 20cm of the quartz tube
Combustion chamber	Last 10cm of the quartz tube
Heat source	Oven
Temperature reading device	Thermo couple

Table 3.1: Essential components of Scenario one.

3. Third step, verify if the analysis in the second step is valid by deciding the flow regime of the gas mixture.
4. Fourth step, calculate the maximum inlet tube for obtaining a homogeneous gas mixture.
5. Fifth step, determine the rising temperature due to combustion and verify if the temperature is safe.

3.1.1 Oven temperature control

As mentioned in previous chapter, the flame quenching problem may happen in small space combustion, for stabilizing flame, the oven temperature should always set above the auto ignition point of the the methane, so the temperature of the oven is decided to be 600C.

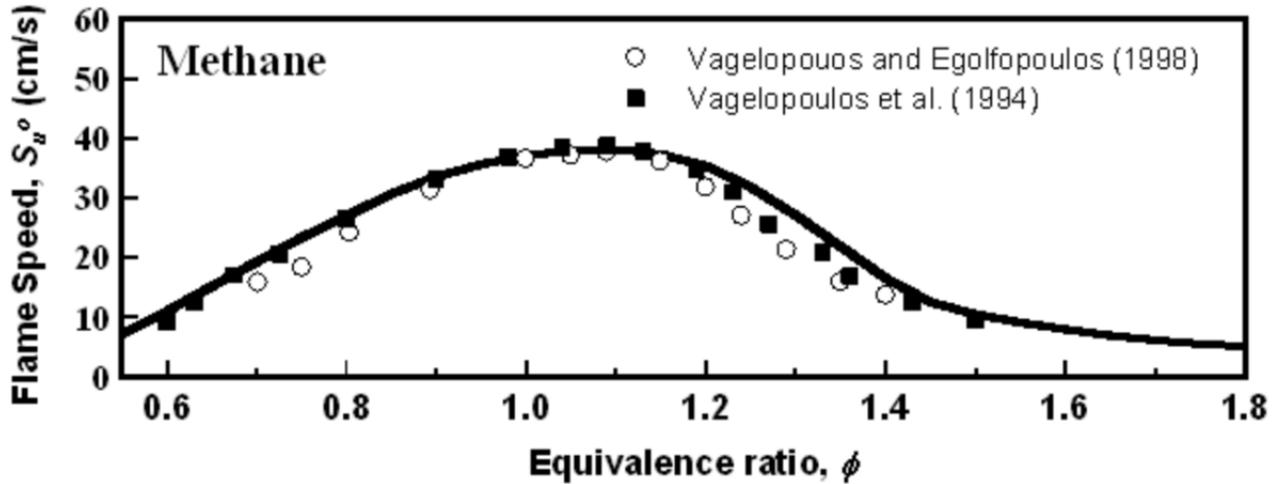


Figure 3.2: Relation between equivalence ratio and flame velocity. [13]

3.1.2 Mass flow control

If the flow velocity is faster than the flame velocity, the flame will be pushed towards the exhaust ending of the tube and finally extinguished. If the flame velocity is faster than gas velocity, the flame will be pushed towards the mixer side which also will lead to the extinguishing of the fire. Therefore, to fix the flame in a stable point inside the tube, the gas velocity should be equivalent to the flame velocity. The flame velocity mainly depends on the equivalence ratio of the air-fuel mixer 3.2: The equivalence ratio which decides the flame velocity is defined as the mass ratio of real gas to oxygen ratio to stoichiometric gas to oxygen ratio:

$$\phi = \frac{\frac{M_{\text{realgas}}}{M_{\text{realair}}}}{\frac{M_{\text{stoichiometricgas}}}{M_{\text{stoichiometricreal}}}} \quad (3.1)$$

. Stoichiometric gas to oxygen ratio is the ideal ratio which represents the ideal situation that the gas fully reacts with the oxidizer without any excess oxygen left. For convince, the equivalence ratio in this design is set to be 1, and then the flame velocity equals 40cm/s . According to 3.1 the real gas to oxygen ratio should equal to stoichiometric gas to oxygen ratio. From 2.12, to burn out one mole of CH_4 , two moles of O_2 are consumed. According to the periodic table [14], the mole mass for CH_4 is 16g/mole , the mole mass for O_2 is 32g/mole . Also the mass ratio of oxygen to air is 23.5% at 600°C [15], which means the stoichiometric methane to air mass ratio equals:

$$\frac{M_{\text{realmethane}}}{M_{\text{realair}}} = \frac{M_{\text{stoichiometricgas}}}{M_{\text{stoichiometricreal}}} = 16\text{g/mole} : 2 \times 32\text{g/mole}/23.5\% = 1 : 17.286 \quad (3.2)$$

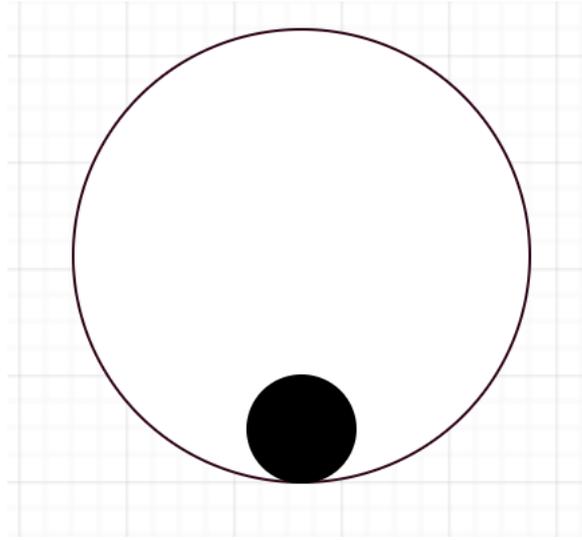


Figure 3.3: The cross section area which the gas can pass through, the large circle represents the silica tube, the black dot stands for the thermo couple

Referring to equation 2.2, the mass flow can be calculated as:

$$\dot{m} = \rho V_{\text{avg}} A_c = \int_{A_c} \rho u(r) dA_c \quad (3.3)$$

The V_{avg} is equivalent to flame velocity. According to empirical result [16], the density of methane-air mixture at stoichiometric methane to air mass ratio is around 0.387 kg/m^3 . The cross section which the gas can pass through is shown as figure 3.3

The area of this cross section equals the cross-section area of the silica tube minus the cross-section area of the thermocouple which equals

$$\left(\frac{5\text{mm}}{2}\right)^2 \times \pi - \left(\frac{1\text{mm}}{2}\right)^2 \times \pi = 18.85 \text{ mm}^2 = 1.89 \times 10^{-5} \text{ m}^2 \quad (3.4)$$

Thus the mass flow of the gas mixture is decided as:

$$\dot{m}_{\text{mixture}} = 0.387 \text{ kg/m}^3 \times 0.4 \text{ m/s} \times 1.89 \times 10^{-5} \text{ m}^2 = 2.93 \times 10^{-6} \text{ kg/s} = 2.93 \times 10^{-3} \text{ g/s} \quad (3.5)$$

Converting this unit to gram per hour:

$$2.93 \times 10^{-3} \text{ g/s} = 8.13 \text{ g/h} \quad (3.6)$$

Taking the 3.2 into consideration, the mass flow for methane is:

$$\dot{m}_{\text{methane}} = \dot{m}_{\text{mixture}} \times \frac{1}{1 + 17.286} = 1.59 \times 10^{-4} \text{ g/s} = 0.44 \text{ g/h} \quad (3.7)$$

The mass flow for air is

$$\dot{m}_{\text{air}} = \dot{m}_{\text{mixture}} \times \frac{17.286}{1 + 17.286} = 2.77 \times 10^{-3} \text{ g/s} = 7.68 \text{ g/h} \quad (3.8)$$

3.1.3 Flow regime decision

To verify if the analysis is valid in section 3.1.2, it is very important to verify if the flow inside the combustion chamber is laminar. As calculated in previous section 3.1.2, the air occupies most part of the gas mixture. Therefore the kinematic and dynamic viscosity of the gas at $600^{\circ}C$ are approximately equivalent to the kinematic and dynamic viscosity of the air at $600^{\circ}C$, which are $5.128 \times 10^{-5}m^2/s$ and $3.017 \times 10^{-5}kg/m$ [17]. Referring to 2.5 the hydraulic diameter of the cross section which the gas can pass through equals

$$D = \frac{4 \times 1.89 \times 10^{-5}m^2}{2\pi\left(\frac{5 \times 10^{-3}m}{2}\right) + 2\pi\left(\frac{1 \times 10^{-3}m}{2}\right)} = 4 \times 10^{-3}m \quad (3.9)$$

So referring to 2.4 the Reynold number could be decided as:

$$Re = \frac{0.4m/s \times 4 \times 10^{-3}m}{5.128 \times 10^{-5}m^2/s} = 31.2 \quad (3.10)$$

which is far away less than 2100, this result shows that the flow inside the combustion chamber is laminar and the analysis in section 3.1.2 is valid.

3.1.4 Maximum inlet tube length for fully mixing

The minimum length requirement for the quartz tube part which is in the outside of the oven is that the length should be long enough for gases fully mixing. After pass through the connector, the methane and air is going to form a homogeneous mixture inside quartz tube. According to 2.10 and the diffusivity of methane in air at room temperature is $0.21 \times 10^{-4}m^2/s$ [18] the maximum pipe length for obtaining a homogeneous gas mixture can be calculated as:

$$L_{\max} = \frac{2.93 \times 10^{-6}kg/s}{0.21 \times 10^{-4}m^2/s \times 0.387kg/m^3 \times \pi} = 0.115m \quad (3.11)$$

Thus in this case 20cm inlet tube is enough for gases to form a homogeneous mixture.

3.1.5 Temperature rise due to combustion

It is crucial to determine the temperature rise caused by burning. As introduced in chapter one, temperature rising involves with the combustion heat, the heat loss due to heat transfer, and the heat capacity of the combustion chamber.

The mass flow for methane is $1.66 \times 10^{-4}g/s$ also equivalent to $1.59 \times 10^{-4}/16 = 0.99 \times 10^{-5}mole/s$, referring to section 2.5.2, the reaction power is

$$P_{\text{reaction}} = 0.99 \times 10^{-5}mole/s \times 802.3kJ/mole = 7.94 \times 10^{-3}kJ/s = 7.94W \quad (3.12)$$

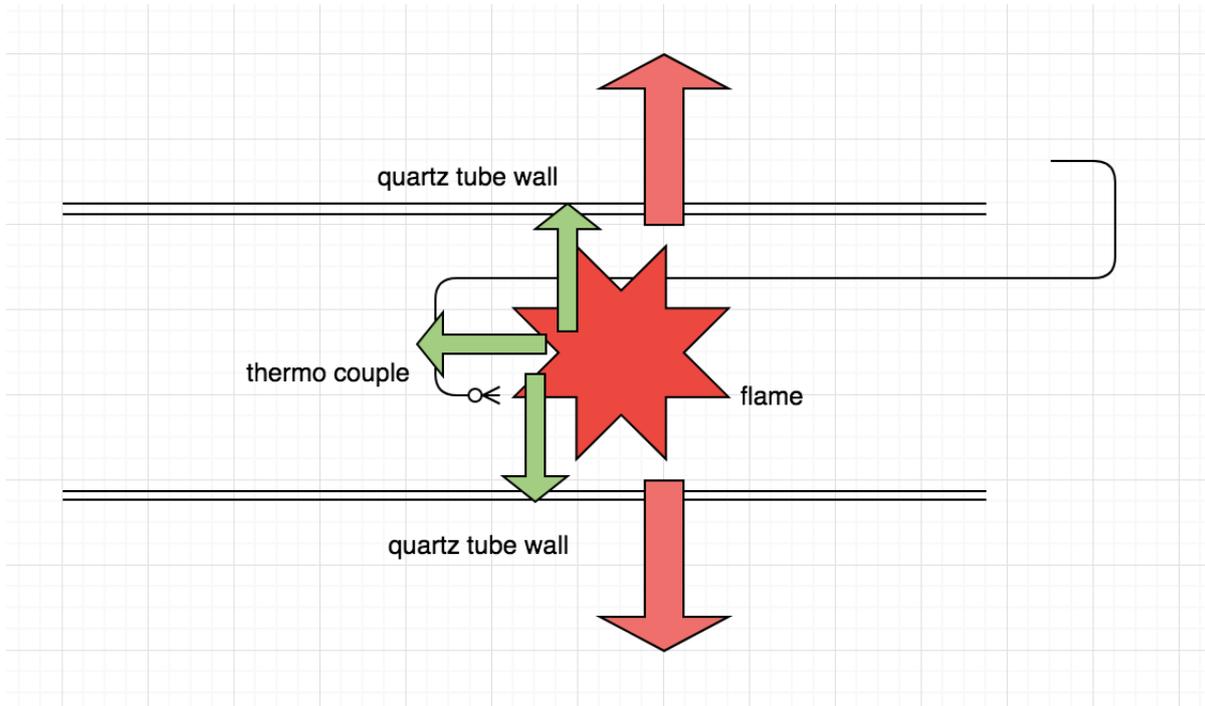


Figure 3.4: Illustration process of heat flow in scenario one, the green arrows shows the direction of the heat which is absorbed by the system, the red arrows show the heat which is transferred to the outside of the quartz tube.

On the other hand, as stated in previous chapter 2.5.2, the temperature rising of the silica tube is related to the heat absorbed by the silica tube also the specific heat. The total heat absorbed by the silica tube is approximately equivalent to the total heat generated by the flame minus the heat absorbed by thermocouple minus the heat transfers to the outside of the tube. Since the flame only happens inside the oven, this heat transfer process only involves the tube part which is inside the oven. The heat flow is indicated as figure 3.4

To determine the final temperature inside the tube easily, the system could be analogous to an electrical system shown as figure 3.5 . The power source is the burning power which is analogous to a current source. The thermocouple and glass tube are analogous to two capacitors which can accumulate the heat power and generate a temperature difference. The thermal conductivity of the glass tube wall which conducts the heat power is analogous to the conductor or inversely the resistor in the electrical circuit. At the start the current charges the capacitors and a tiny voltage difference is rising, this tiny voltage difference will give a current through the resistor, like the temperature difference will lead to a heat transfer between the system and outside environment. The voltage keeps on increasing for a while until the current through the resistor equals the source current. Then the current source

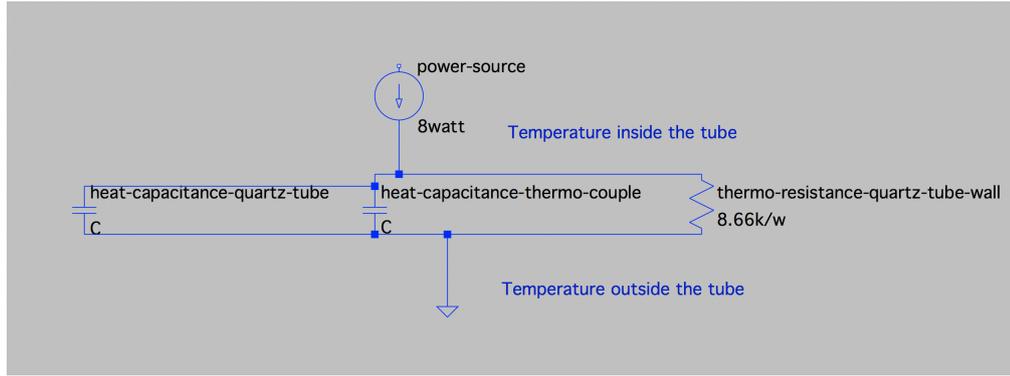


Figure 3.5: Analogous electrical circuit in scenario one

stops to charge the capacitor and the voltage would not rise any more. As the system equals thermal equilibrium state, the temperature would not rise any more. The voltage difference, in this case, is calculated as the source current times the resistance of the resistor. Identically the temperature difference is calculated as the source power multiply with the thermoresistance,

Inside the oven this is almost no wind, so the convection effect inside the oven is small enough to be ignored, the radiation effect is such small that can be neglected as well, so only the conduction will influence the heat transfer. The thermal resistance is calculated as equation 2.19. The tube surface wall involves conduction is only the part inside the oven which is $0.1m$ long, the conductivity of the glass is $1.05W/m \cdot K$ at $600^{\circ}C$ [19], the inner diameter of the tube $2.5mm$ and outer wall diameter is $3.5mm$. Therefore the thermal resistance of the quartz tube wall is estimated by:

$$R = \frac{1}{\frac{3.5mm-2.5mm}{4 \times \pi \times 3.5mm \times 2.5mm \times 1.05W/m \cdot k}} = 8.66K/W \quad (3.13)$$

The power generated by the flame is approximately $8J/s$. Thus the temperature difference is calculated as:

$$\Delta T = R \times P = 8.66K/W \times 8W = 69.28K = 69.28^{\circ}C \quad (3.14)$$

Since the temperature outside the oven is $600^{\circ}C$, the temperature inside the tube is

$$T = T_{\text{inside}} + \Delta T = 600^{\circ}C + 69.28^{\circ}C = 669.28^{\circ}C \quad (3.15)$$

Which means the thermocouple should detect a temperature around $669.28^{\circ}C$ if the gas is fully reacted. This temperature is much smaller than the glass melting temperature $1500^{\circ}C$ [20], the experiment would not take a risk at this case.

3.1.6 Conclusion of scenario one

In short, for generating a stable flame of scenario one, there are several requirements:

- The oven temperature should be set around $600^{\circ}C$.
- The mass flow value of methane is $0.44g/h$.
- The mass flow value of air is $7.68g/h$.

Once all the requirements are fulfilled, a stable flame is created, and the thermocouple will detect a temperature around $669.28^{\circ}C$.

3.2 Scenario two: Chip-based burning method

The scenario two is designed to realize micro-scale combustion on a microfabricated chip. As shown in figure 3.6, the combustion chamber is made of a fabricated chip. The chip consists of 4 layers. A silicon layer with thickness $525\mu m$ is put between two silicon nitride layers with thickness $2\mu m$. One side of silicon nitride is stuck to a platinum layer for the sake of heating and temperature sensing. A cavity is made and etching from the other side of the silicon nitride layer. A glass wafer sticks to this side of the layer to seal the cavity. Two holes are drilled on the glass wafer to put in the inlet tube and outlet tube. The methane and air are mixed in the inlet tube and get into the cavity. The essential components of scenario two are revealed in table 3.2. The mixture reacts inside the cavity and exhaust gas emit out via the outlet tube. The design procedures are:

1. First step, determine how to create the combustion chamber.
2. Second step, decide the chamber temperature and how to control the temperature.
3. Third step, calculate the mass flow values of both methane and air which are needed to stabilize the flame inside the chamber.
4. Fourth step, verify if the analysis in the third step is valid by deciding the flow regime of the gas mixture inside the combustion chamber.
5. Fifth step, determine the length requirement for inlet tube.
6. Sixth step, determine the rising temperature due to combustion and verify if the temperature is safe.

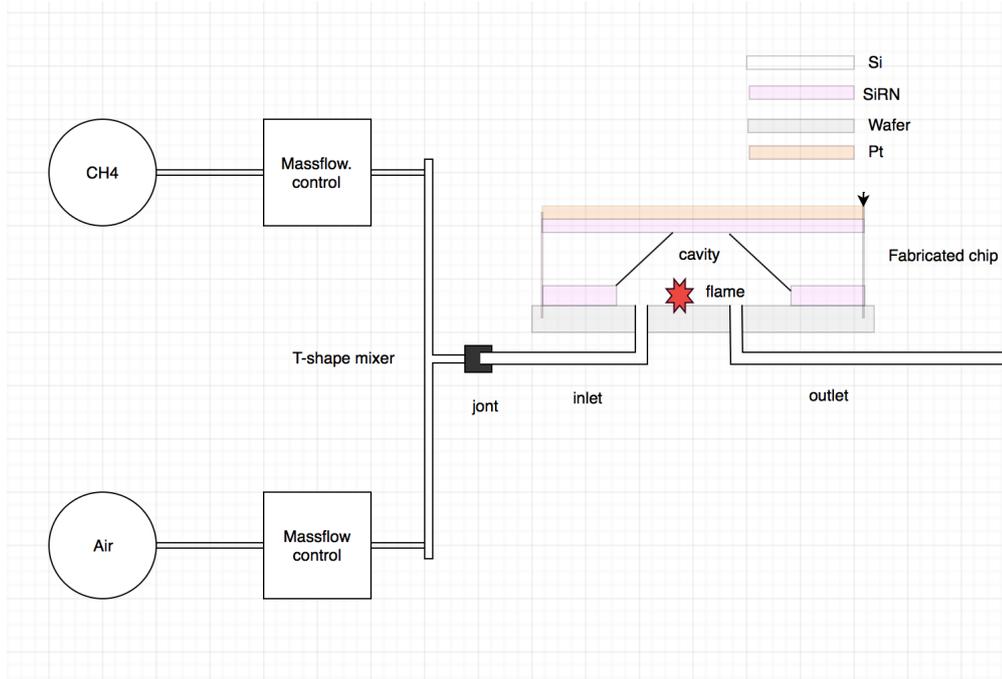


Figure 3.6: Schematic of quartz tube burning method

Combustion chamber	a cavity created on the silicon layer of the chip
Heat source	platinum wires fabricated on the platinum layer of the chip
Temperature reading device	platinum wires fabricated on the platinum layer of the chip

Table 3.2: Essential components of Scenario two.

3.2.1 Chamber temperature control

The platinum layer consists of platinum wires which can be used as heaters and temperature sensors. The platinum spiral heater heats the combustion chamber till 600°C which is beyond the ignition point of methane in air. This temperature is monitored by the platinum spiral sensor as well.

3.2.2 Combustion chamber design

The cavity is created by potassium hydroxide etching and sealing by a piece of Borofloat-33 glass. Two holes are drilled on the glass to put the inlet and outlet tube.

The potassium hydroxide (KOH) which etches the silicon at very large different rate at $\langle 100 \rangle$ and $\langle 111 \rangle$ directions serves as the etchant in this fabrication process. This process starts with etching the silicon through a rectangle hole in the mask material. As shown in figure 3.7, the KOH etches 400 times faster in $\langle 100 \rangle$ direction than in $\langle 111 \rangle$ direction [22]. Thus according to this relation, the

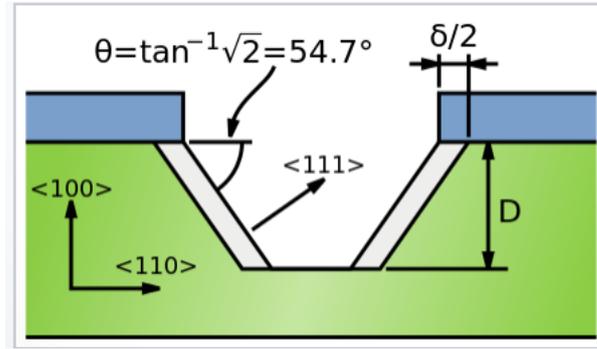


Figure 3.7: Demonstration of KOH etching on silicon [21]

angle between $\langle 111 \rangle$ side wall and the mask surface could be described by the expression:

$$\theta = \tan^{-1}\sqrt{2} = 54.7^\circ \quad (3.16)$$

The figure 3.8 shows a front view of the chip. The green layer is the platinum. Spiral A to E can be used as joule heating wire or temperature sensor. Between the platinum layer and cavity is a silicon nitride layer. Since the thickness of the silicon is too thin, if the length of the back side of the cavity is too long, the silicon nitride is very brittle. So only spiral B C D is decided to be used. The dark purple rectangle indicates the backside of the cavity, which the area is $2600\mu m \times 6600\mu m$ and just covered the spiral B C D. Since the thickness of the silicon layer is $525\mu m$, and according to 3.16, the dimension of the front view of the cavity could be calculated as:

$$width = 2600\mu m + 525\mu m \times \tan(54.7^\circ) = 3344\mu m \quad (3.17)$$

$$length = 6600\mu m + 525\mu m \times \tan(54.7^\circ) = 7344\mu m \quad (3.18)$$

The light purple rectangle shows the frontside area of the mask.

3.2.3 Mass flow control

The light purple rectangle shows the frontside area of the mask. As introduced in section 3.3.3, for the sake of stabilizing the flame in combustion chamber, this situation is demonstrated in figure 3.9. The flame velocity should equal flow velocity. When equivalence ratio of gas mixture equals one, the flame velocity equals $40cm/s$, so the flow velocity should equal $40cm/s$ as well. The mass flow value of the gas mixture is related to the area of the cross-section, the flow velocity and the gas density at $600^\circ C$. Since the flame velocity and air-methane mixture density at $600^\circ C$

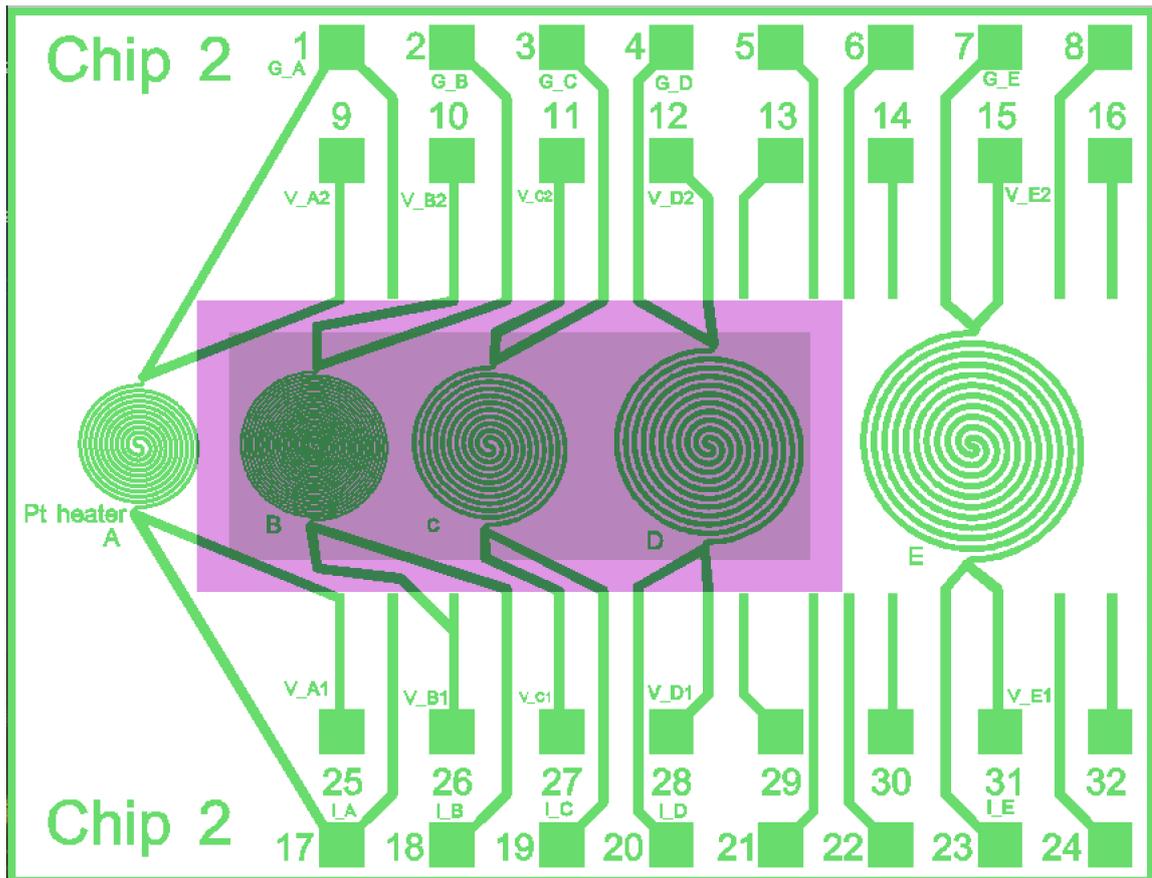


Figure 3.8: Illustration of mask design, the green texture shows the spirals and connections fabricated on platinum layer, the dark purple rectangle shows the backside of the cavity and the light purple shows the frontside of the cavity.

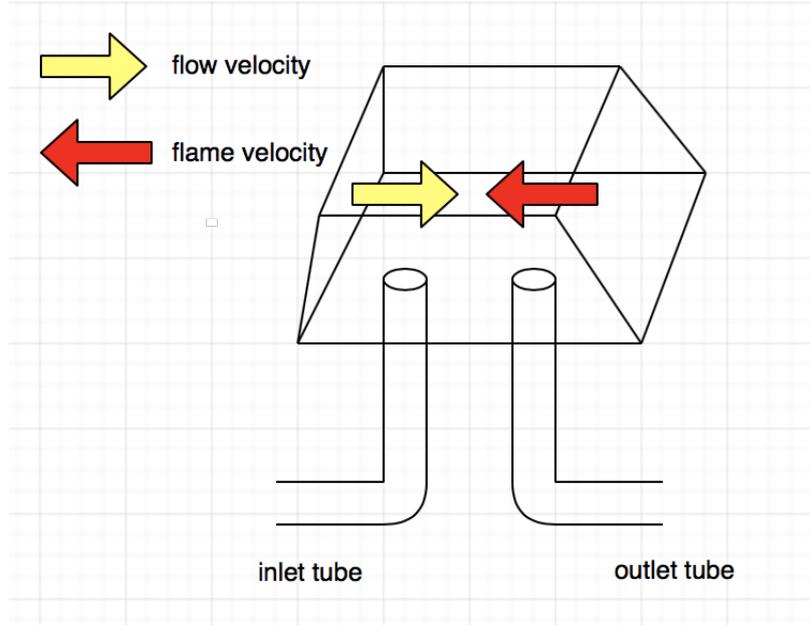


Figure 3.9: Demonstration of flame stabilization in the combustion chamber of scenario two.

are already known in section 3.3.3, the only thing needed to calculate is the cross-section area. According to the section 3.2.2, the cross-section of the combustion chamber is a trapezoid with bottom length $2600\mu m$ and $3344\mu m$ respectively, the height of the trapezoid is equivalent to the thickness of the silicon layer. Therefore the cross-section area equals:

$$A_c = \frac{(2600\mu m + 3344\mu m) \times 525\mu m}{2} = 1.56 \times 10^{-6} m^2 \quad (3.19)$$

Referring to equation 2.2, the mass flow of the gas mixture is determined as:

$$\dot{m} = 1.5610^{-6} m^2 \times 0.4 m/s \times 0.387 kg/m^3 = 0.24 mg/s = 2.4 \times 10^{-4} g/s = 869.4 mg/h \quad (3.20)$$

Taking the relation 3.2 into consideration, the mass flow for methane is:

$$\dot{m}_{\text{methane}} = \dot{m}_{\text{mixture}} \times \frac{1}{1 + 17.286} = 1.32 \times 10^{-5} g/s = 47.56 mg/h \quad (3.21)$$

The mass flow for air is

$$\dot{m}_{\text{air}} = \dot{m}_{\text{mixture}} \times \frac{17.286}{1 + 17.286} = 821.84 mg/h \quad (3.22)$$

3.2.4 Flow regime decision

To verify if the analysis is valid in section 3.2.3, it is very important to verify if the flow inside the combustion chamber is laminar. As calculated in previous section

3.2.3, the air occupies most of the gas mixture. Thus the kinematic and dynamic viscosity of the gas at $600^{\circ}C$ are approximately equivalent to the kinematic and dynamic viscosity of the air at $600^{\circ}C$, which are $5.128 \times 10^{-5} m^2/s$ and $3.017 \times 10^{-5} kg/m$. Referring to 2.5 the hydraulic diameter of the cross-section which the gas can pass through equals four times the cross-section area divided by the circumference of the trapezoid shape cross-section.

$$D = \frac{4 \times 1.56 \times 10^{-6} m^2}{646 \mu m \times 2 + 3344 \mu m + 2600 \mu m} = 8.624 \times 10^{-4} m \quad (3.23)$$

so as Indicated by 2.4 the Reynold number could be decided as:

$$Re = \frac{0.4 m/s \times 8.624 \times 10^{-4} m}{5.128 \times 10^{-5} m^2/s} = 6.73 \quad (3.24)$$

which is far away less than 2100, this result shows that the flow inside the combustion chamber is laminar, the analysis in section 3.2.3 is valid.

3.2.5 Maximum inlet tube length

The minimum length requirement for the inlet tube is decided by the minimum length for obtaining a homogeneous mixture. This value is calculated at room temperature which is $23^{\circ}C$, the mixture density under this temperature is approximately $1.1 kg/m^3$ [23]. Thus the the maximum inlet tube length for obtaining a homogeneous gas mixture can be calculated as:

$$L_{\max} = \frac{2.4 \times 10^{-7} kg/s}{0.21 \times 10^{-4} m^2/s \times 1.1 kg/m^3 \times \pi} = 3.3 \times 10^{-3} m \quad (3.25)$$

Therefore the inlet tube length is at least $3.3 \times 10^{-3} m$.

3.2.6 Temperature rise due to combustion

The temperature rising can be calculated as in the scenario one. The mass flow for methane is $1.32 \times 10^{-5} g/s$ also equivalent to

$$1.32 \times 10^{-5} g/s / 16 g/mole = 8.26 \times 10^{-7} mole/s \quad (3.26)$$

Referring to section 2.5.2, the reaction power is

$$P_{\text{reaction}} = 8.26 \times 10^{-7} mole/s \times 802.3 kJ/mole = 0.67 \times 10^{-3} kJ/s = 0.67 W \quad (3.27)$$

The heat flow is shown as in figure 3.10. The red arrows indicate the heat transfer to the outside of the combustion chamber. The green arrows show the heat absorbed by the glass wafer and the silicon wall. Similarly. This heat transfer sys-

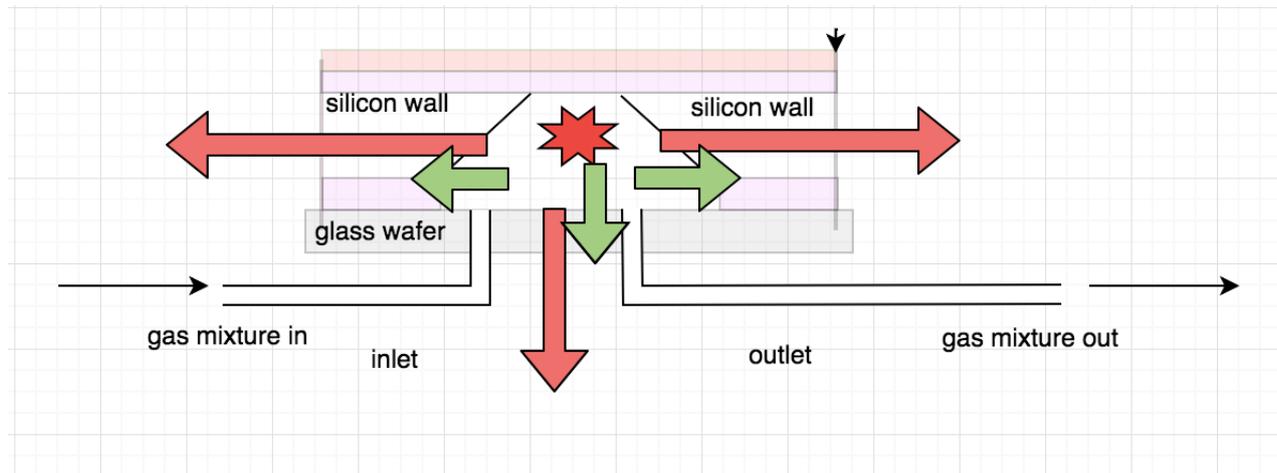


Figure 3.10: Illustration process of heat flow in scenario two, the green arrows shows the direction of the heat which is absorbed by the system, the red arrows show the heat which is transferred to the outside of the combustion chamber.

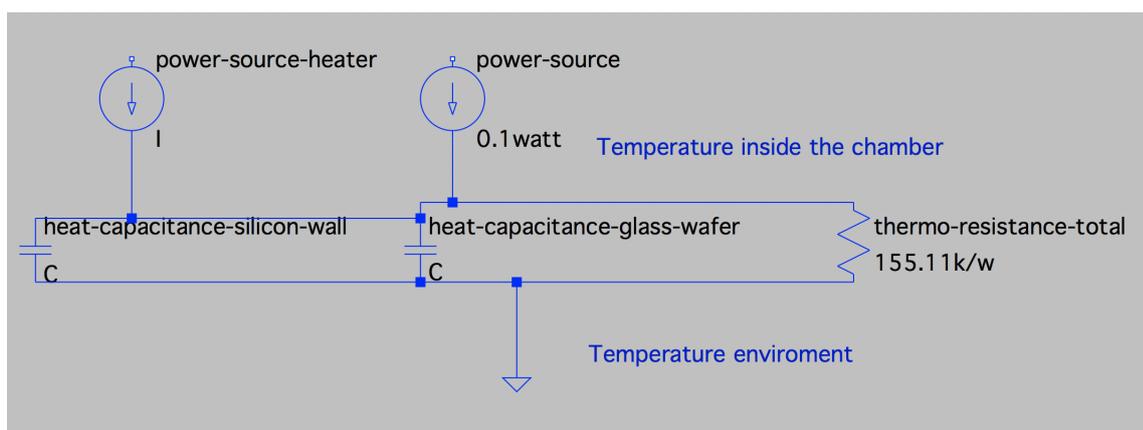


Figure 3.11: Analogous electrical circuit in scenario one

tem is analogous to a electrical system as shown in figure 3.11 . At the start, the power source of the heater heated up until the chamber temperature stable at $600^{\circ}C$ and maintain a thermal equilibrium state. The environment temperature is around $23^{\circ}C$, and the temperature difference is around $577^{\circ}C$. After that, the reaction heat starts to charge the heat capacitance of the silicon wall and the glass wafer, which generate more temperature difference between the combustion chamber and the environment. This temperature difference exerts on the thermal resistance and makes more heat power transfers from the chamber to the environment. When this power equals the power generated by the reaction, the system reaches thermal equilibrium again, and the temperature difference is decided as the reaction power times the thermal resistance.

The total thermal resistances consists of the thermal resistances of the silicon wall and the glass wafer. Conduction mostly dominates the heat transfer. Since the silicon wall is not perfectly straight, then the distance between the silicon wall is taken the average value. The thermal conductivity of silicon is $62W/m \cdot K$ [24]. Thus the thermal resistance of the silicon wall along the horizontal direction is

$$R_1 = \frac{2381\mu m}{62W/m \cdot K \times 3344\mu m \times 525\mu m} + \frac{3821\mu m}{62W/m \cdot K \times 3344\mu m \times 525\mu m} \quad (3.28)$$

The thermal resistance of the silicon wall alone vertical direction (referring to the top view of the chip 3.8) is

$$R_2 = \frac{3582\mu m}{62W/m \cdot K \times 7744\mu m \times 525\mu m} \times 2 \quad (3.29)$$

The total thermal resistance of the silicon wall

$$R_{\text{siliconwall}} = R_1 + R_2 = 99.61K/W \quad (3.30)$$

For the glass wafer, the thickness is approximately $1.1mm$, the thermal conductivity is $1.2W/K \cdot m$ [25]and the area equals the front side area of the cavity, so the thermal resistance of the glass wafer is

$$R_{\text{glasswafer}} = \frac{1.1mm}{6600\mu m \times 2600\mu m \times 1.2W/K \cdot m} = 53K/W \quad (3.31)$$

The entire thermal resistance of the combustion chamber is

$$R = R_{\text{siliconwall}} + R_{\text{glasswafer}} = 155.11K/W \quad (3.32)$$

Therefore the temperature inside the chamber after combustion approximately equals the sum of the initial temperature and the temperature rise due to the reaction.

$$T = R \times P + T_{\text{initial}} = 155.11K/W \times 0.67W + 600^{\circ}C = 704^{\circ}C \quad (3.33)$$

This temperature is much lower than the melting point of any chip material. Therefore the chip won't break down.

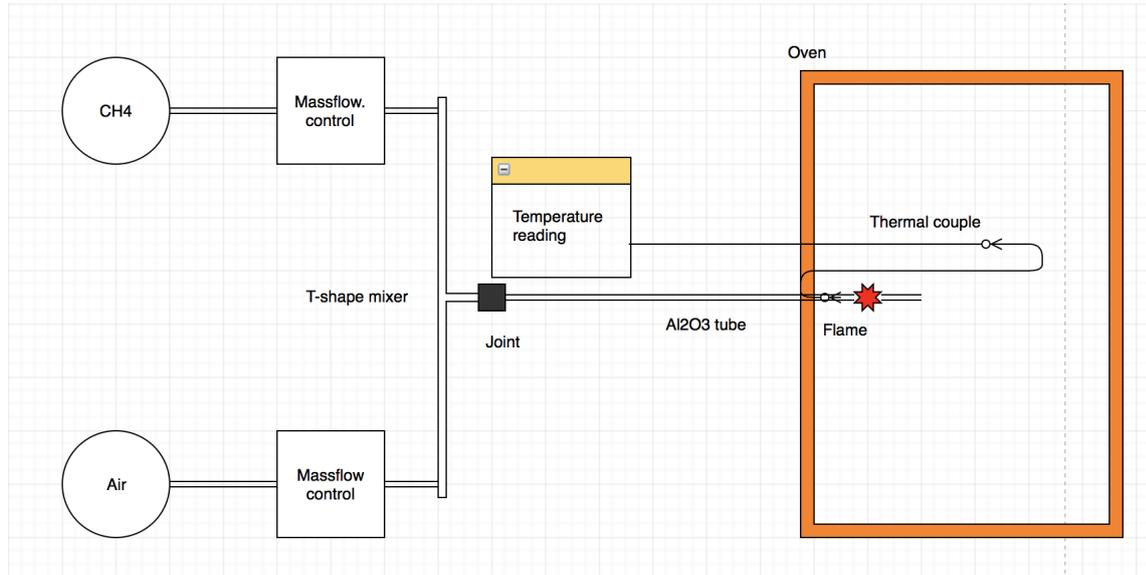


Figure 3.12: the schematic of scenario three

3.2.7 Conclusion of scenario two

In short, for generating a stable flame of scenario one, there are several requirements:

- The combustion chamber should be heated up to around $600^{\circ}C$.
- The inlet tube length is at least $3.3 \times 10^{-3}m$.
- The mass flow value of methane is $47.56mg/h$.
- The mass flow value of air is $821.84mg/h$.

Once all the requirements are fulfilled, a stable flame is created, and the temperature around $704^{\circ}C$ will be detected.

3.3 Scenario three: 3D printed aluminum oxide tube

In principle varieties shapes of combustion chambers can be fabricated by 3D printing. The scenario three is based on scenario one, which substitutes the quartz tube with a 3D printed aluminum oxide tube. also instead of inserting the thermocouple inside the tube, a thermocouple is stuck to the outside wall of the tube at the area where the flame probably happens. The shape of the aluminum oxide tube is slightly different from the quartz tube,

This $80cm$ long tube is divided into two parts. The first part is an ordinary circular tube with length $40cm$, inner diameter $0.5mm$, and wall thickness $0.1mm$. This part

is put outside of the combustion chamber and design for getting a homogeneous mixture. The second part is a changing diameter tube and serves as the combustion chamber. The start of the second part is a circle with diameter 0.5mm and wall thickness 0.1mm , and the end of the second part is a circle with diameter 1mm and wall thickness 0.1mm . The outlook of the tube is like a trumpet and shown as figure 3.13. In comparison with scenario one, there are mainly two advantages. The first one is aluminum oxide tube can withstand a higher temperature than the silicon oxide. The second one is it's easier to stabilize the flame. In scenario one, even though theoretically it is possible to balance the gas flow velocity and flame velocity, but in reality, there are some problems to make the flame unstable such as it's hard to make sure the component of the gas mixture is as precise as designed. Also, the density of the gas mixture is not precise which will lead to a result that the flow velocity is not balanced with the flame velocity. However, since the speed of the gas keeps on increasing towards the start of trumpet starting, which means even there are some derivations of velocity from the ideal situation, there must be a stable point inside the tube.

On the other hand, due to fabrication limitation, the wall thickness might not be allowed to be less than 0.5mm . Therefore, the possibility for this scenario is going to be investigated with the 3D printing company.

Some essential components are revealed in the table 3.3, and the schematic of scenario three is revealed in the 3.12, design procedures of scenario three are:

1. First step, decide the oven temperature for maintaining continuous combustion.
2. Second step, give a default combustion power, verify if the power is safe by calculating the temperature rise due to the burning.
3. Third step, If the power is safe, calculated the mass flow for both methane and air.
4. Fourth step, calculate the maximum inlet tube length for getting a homogeneous mixture.
5. Fifth step, find the possible flame stable point inside the combustion chamber
6. Sixth step, verify if the analysis in the fifth step is valid by deciding the flow regime inside the combustion chamber.

3.3.1 Oven temperature control

Same reasoning as mentioned in scenario one 3.1.1. For stabilizing flame, the oven temperature should always set above the autoignition point of the methane, so the

Inlet tube	First 40cm of the 3D printing tube
Combustion chamber	Last 40cm of the 3D printing tube
Heat source	Oven
Temperature reading device	Thermo couple

Table 3.3: Essential components of Scenario one.

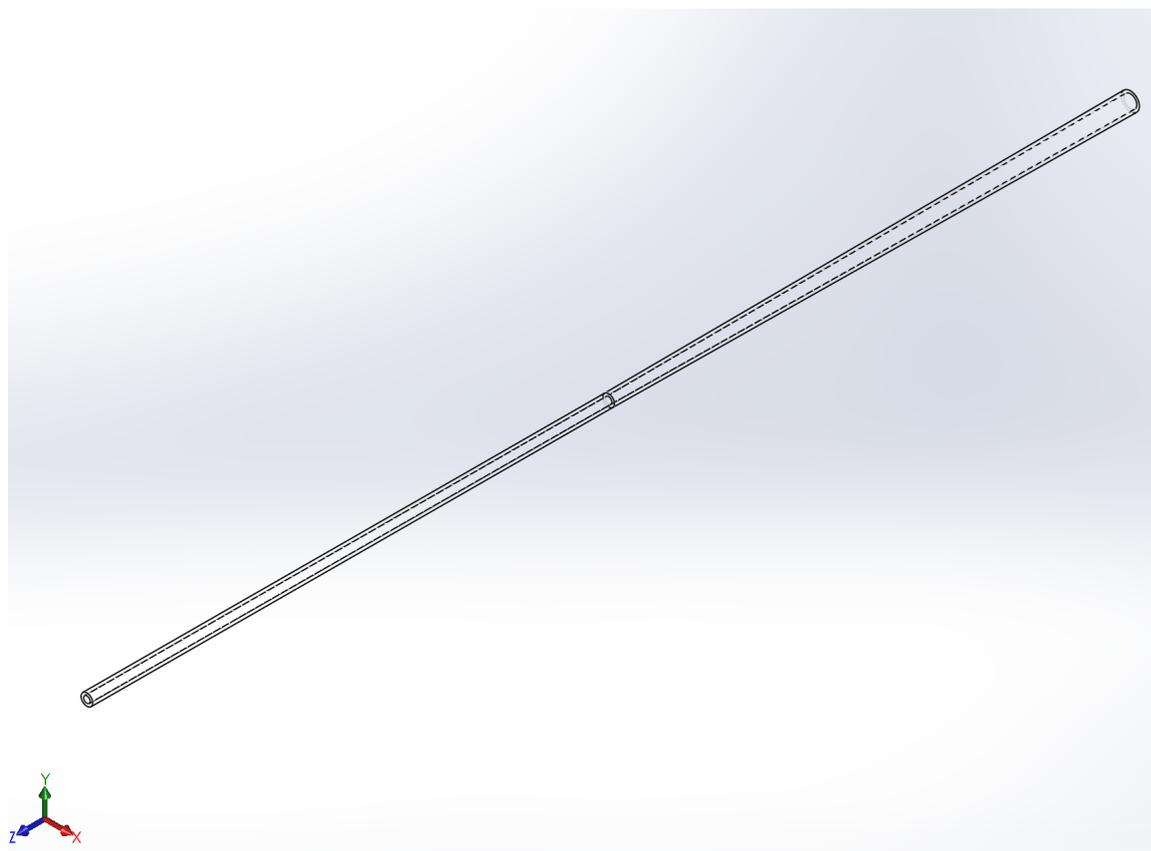


Figure 3.13: the outlook of aluminum oxide tube

temperature of the oven is decided to be $600^{\circ}C$.

3.3.2 Combustion temperature

In this scenario, the combustion power is first assumed to be $0.1W$ then verify if the power is dangerous to the combustion chamber by calculating the temperature rise due to combustion. As calculated in previous sections, the temperature rise due to combustion can be calculated as the combustion power multiplies with the resistance. In this case, the conductivity of aluminum oxide at $600^{\circ}C$ is $12W/m$ [26]. So similarly, the thermal resistance for is calculate as:

$$R = \frac{1}{\frac{0.1mm}{4 \times \pi \times 0.356mm \times 0.256mm \times 12W/m \cdot k}} = 7.28K/W \quad (3.34)$$

The temperature difference is thus:

$$\Delta T = R \times P = 7.28/W \times 0.1W = 0.8^{\circ}C \quad (3.35)$$

So the temperature of the combustion chamber after combustion is around $600.8^{\circ}C$ which is far away below the melting point of aluminum oxide $2054^{\circ}C$. Therefore $0.1W$ is a safe power.

3.3.3 Mass flow control

According to section 3.3.2, $0.1W$ is a safe power. For generating $0.1W$ power, the mass flow of methane is $1.932 \times 10^{-6}g/s = 6.92 \times 10^{-3}g/h$, and if the equivalence ratio equals 1, the mass flow of air and gas mixture are $3.52 \times 10^{-5}g/s = 1.27 \times 10^{-1}g/h$ and $3.72 \times 10^{-5}g/s = 1.34 \times 10^{-1}g/h$

3.3.4 Maximum tube length for fully mixing

The minimum length requirement for the inlet tube is decided by the minimum length for obtaining a homogeneous mixture, this value is calculated at room temperature which is $23^{\circ}C$, the mixture density under this temperature is approximately $1.1kg/m^3$. Thus the the maximum inlet tube length for obtaining a homogeneous gas mixture can be calculated as:

$$L_{\max} = \frac{3.52 \times 10^{-8}kg/s}{0.21 \times 10^{-4}m^2/s \times 1.1kg/m^3 \times \pi} = 5.12 \times 10^{-4}m \quad (3.36)$$

So $40cm$ inlet tube is enough for obtaining a homogeneous mixture.

3.3.5 Flame stable point

Calculating the stable flame point is crucial to the place where the thermocouple should stick to.

The maximum flow speed is happened at the entrance of second part which is

$$V_{\max} = \frac{3.52 \times 10^{-8} \text{kg/s}}{0.387 \text{kg/m}^3 \times \left(\frac{1 \times 10^{-3} \text{m}}{2}\right)^2 \times \pi} = 12 \text{cm/s} \quad (3.37)$$

The minimum flow speed is happened at the end of second part which is

$$V_{\min} = \frac{3.52 \times 10^{-8} \text{kg/s}}{0.387 \text{kg/m}^3 \times \left(\frac{5 \times 10^{-4} \text{m}}{2}\right)^2 \times \pi} = 46 \text{cm/s} \quad (3.38)$$

Thus the stable point which the mixture velocity equals 40cm/s should be inside the tube. when the velocity equals 40cm/s , the cross section area of this point is

$$A = \frac{3.52 \times 10^{-8} \text{kg/s}}{0.387 \text{kg/m}^3 \times 0.4 \text{m/s}} = 2.27410^{-7} \text{m}^2 \quad (3.39)$$

Therefore the diameter of the cross-section area is

$$D = 2 \times \sqrt{\frac{2.27410^{-7} \text{m}^2}{\pi}} = 0.53 \text{mm} \quad (3.40)$$

Since the diameter changes linearly with the length, thus the distance from the flame point to the start of the second part of the tube is

$$L = 40 \text{cm} - \frac{0.53 \text{mm}}{1 \text{mm}} \times 40 \text{cm} = 18.8 \text{cm} \quad (3.41)$$

So the thermocouple should be stick to the point which is 18.8mm far away from the start point of the second part of the tube.

3.3.6 Flow regime decision

To verify if the analysis is valid in section 3.3.5, it is very important to verify if the flow inside the combustion chamber is laminar. As calculated in previous section 3.2.3, the air occupies most of the gas mixture. Thus the kinematic and dynamic viscosity of the gas at 600°C are approximately equivalent to the kinematic and dynamic viscosity of the air at 600°C , which are $5.128 \times 10^{-5} \text{m}^2/\text{s}$ and $3.017 \times 10^{-5} \text{kg/m}$. Since the cross-section of the combustion chamber is a circle, the hydraulic diameter is equivalent to the diameter. As Indicated by 2.4 the Reynold number is proportional to the diameter, the largest Reynold number is calculated at the exit of the combustion chamber with diameter 1mm , which is:

$$Re = \frac{0.4 \text{m/s} \times 1 \times 10^{-3} \text{m}}{5.128 \times 10^{-5} \text{m}^2/\text{s}} = 7.8 \quad (3.42)$$

The largest Reynold number is far away less than 2100, which means the flow inside anywhere of the combustion chamber is laminar, the analysis in section 3.3.5 is valid.

3.3.7 Conclusion of scenario three

In short, for generating a stable flame of scenario three, there are several requirements:

- The combustion chamber should be heated up to around $600^{\circ}C$.
- The mass flow value of methane is $6.92 \times 10^{-3}g/h$.
- The mass flow value of air is $1.27 \times 10^{-1}g/h$.

Once all the requirements are fulfilled, a stable flame is created, and the temperature around $600.8^{\circ}C$ will be detected.

Measurement results

4.1 General introduction

Regarding the fact that preparing the devices and equipment of scenario one may take some time, on the other hand, some chips designed in scenario two were already fabricated before, also the assignment has to be done within ten weeks, the scenario two was finally decided to be realized. All the technical requirements were set up accurately as described in chapter 3.2¹

The devices were set up as figure 4.2. Besides all the devices mentioned in figure 3.6, an additional protection box was put to prevent explosion. Also, a vacuum pump was connected to the protection box through a pipe. The spiral B, and D of the chip which are shown in figure 3.8 were used as the temperature sensor. Spiral C was used as the heater to heat up the combustion chamber. Spiral D and two high voltage suppliers controlled C, and the lab-view board controlled spiral B. Each spiral was connected to four wires which measure the voltage and current through those wires. The voltage and current information were imported to the Lab-view system in the computer. The Lab-View can calculate the resistance and power according to the voltage and current, and convert the resistances to temperature, the gas mixture flows in the direction from spiral D to B.

In this chapter, three experiments for different goals were conducted subsequently.

¹Since the experiment had been done before chapter 3.2 got revised, during the revising process, the mass flow has been changed. Therefore the mass flow values used in the experiment are different from in chapter 3.2.

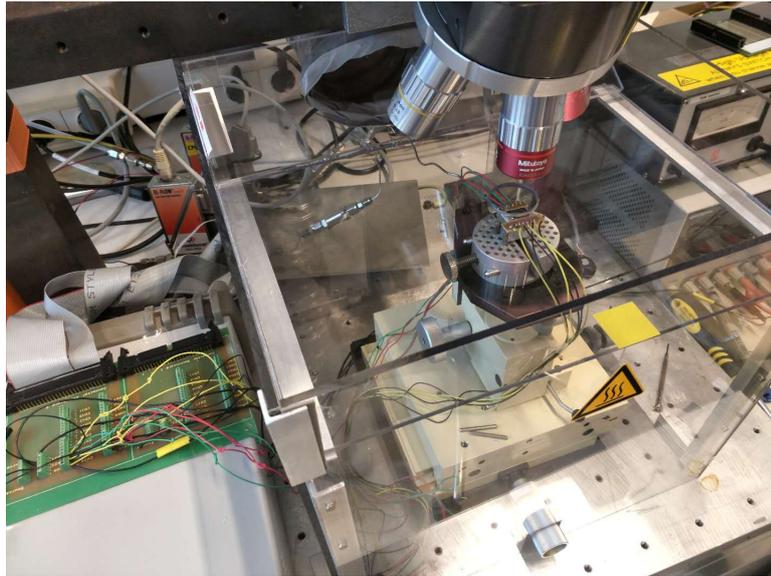


Figure 4.1: Overview of the experiment device

Spiral names	Functions	Controlled by
Spiral B	Temperature sensor to detect chamber temperature	Lab-view board
Spiral C	Heater to heat up whole chamber	Lab-view board
Spiral D	Temperature sensor to detect chamber temperature	High voltage supply

Table 4.1: Functions and controller for each spiral.

4.2 Experiment one

The goal for experiment one is to investigate the sensitivity of the heat sensor spiral B and D also the influence on the heat sensors when adding only the air flow.

The operation process of experiment one is indicated in table 4.2, the 'Operation time' stands for how long did this operation last, the 'Heater temperature' represents how was the temperature of the heater controlled, the 'Flow' shows how was the flow controlled. The result of experiment one is indicated in figure exp1.

According to the result, when there was no flow given, the temperature of spiral D was always higher than spiral B. It is most probably because the spiral D is closer to spiral C than spiral B. Also, it's not hard to observe that when the heater temperature rose, the sensor could detect temperature increasing immediately. However, the fluctuation of sensed value is way too large to decide the precise temperature. On the other hand, first giving then closing the air flow for a while, the temperature of spiral B would first rose then dropped, the temperature of spiral D would first fell then rose. During this process, the temperature lines of sensor B and D formed a 'mouth shape.' Also, the temperature of heater C decreased a little bit. It is because the

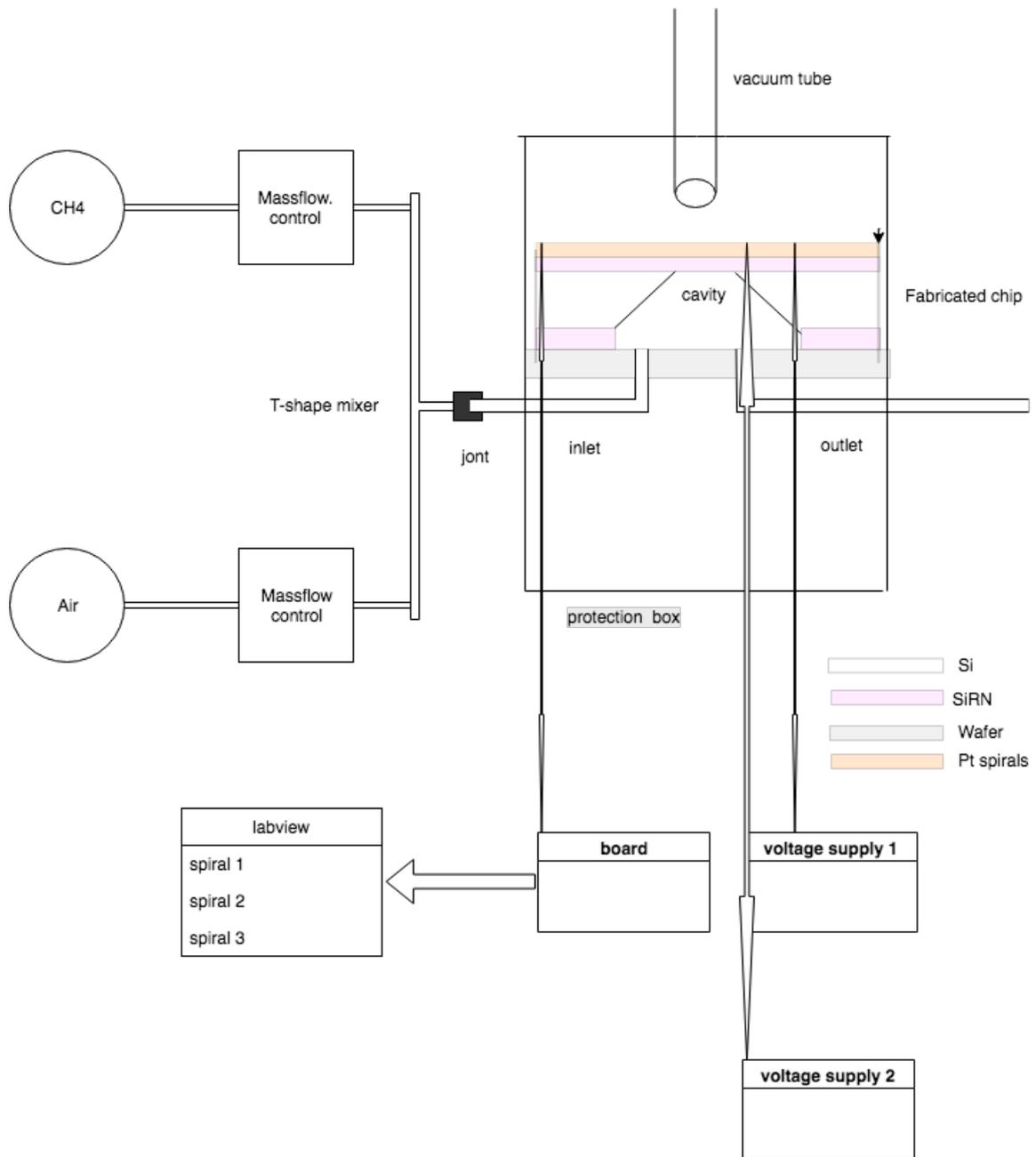


Figure 4.2: Block diagram of the experiment devices set up

Operation time	Heater temperature	Flow
800s	heat up to and keep 50°C	no flow given
4300s	heat up to and keep 110°C	no flow given
650s	keep 110°C	air with mass flow 134mg/h
650s	keep 110°C	no flow given
300s	heat up to and keep 210°C	no flow given
150s	keep 210°C	air with mass flow 134mg/h
150s	keep 210°C	no flow given
300s	keep 210°C	no flow given

Table 4.2: The operation process of experiment one

air first flowed from spiral D, which cooled down D's temperature then took the heat from spiral C to B which decreased C's temperature and increased B's temperature.

Therefore, from the result, some conclusions could be drawn:

- Distance between the heater and sensor would influence the sensor temperature.
- Both spiral B and D can only be treated as indicators of temperature change because they are not able to read the precise temperature value.
- Adding the only airflow would increase the temperature of spiral B and decrease the temperature of spiral and D. Also, the heater temperature would decrease a little bit.
- There is no leakage in the combustion chamber.

4.3 Experiment two

The goal for experiment two is to investigate if the heater can heat up the whole combustion chamber to 600°C, and what is the influence on the heat sensors when adding only the air flow in the high-temperature situation.

The operation process of experiment one is indicated in table 4.3, the 'Operation time' stands for how long did this operation last, the 'Heater temperature' represents how was the temperature of the heater controlled, the 'Flow' shows how large the flow applied. The result of experiment two is indicated in figure 4.8.

According to the result, giving the flow would lead to a consequence that the temperature lines of spiral B and D formed a 'mouth shape' as in experiment one. And when larger flow was given, the 'mouth' opened even larger because larger mass

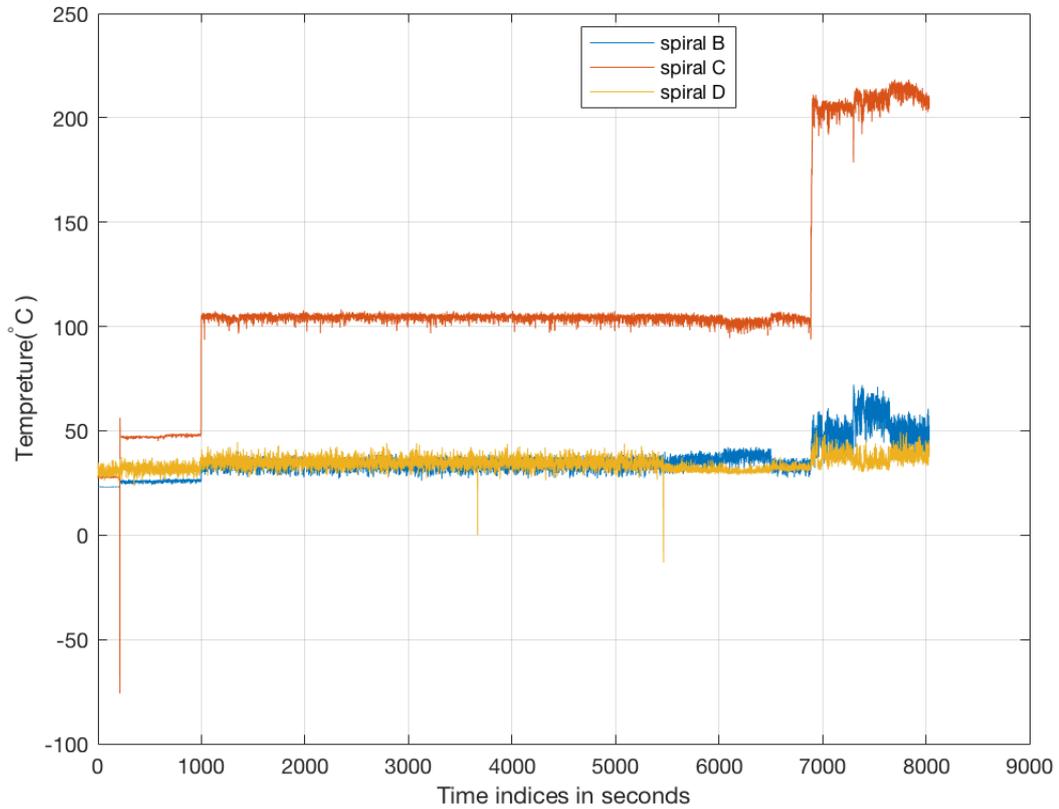


Figure 4.3: the result of experiment one

Operation time	Heater temperature	Flow
2500s	gradually heat up to 520°C	no flow given
600s	keep 520°C	no flow given
600s	keep 520°C	air with mass flow 134mg/h
600s	keep 520°C	no flow given
600s	keep 520°C	air with mass flow 500mg/h
1200s	keep 520°C	no flow given
1200s	heat up and keep to 550°C	no flow given
600s	keep 550°C	air with mass flow 134mg/h
600s	keep 550°C	no flow given
600s	keep 550°C	air with mass flow 500mg/h
1200s	keep 550°C	no flow given

Table 4.3: The operation process of experiment two

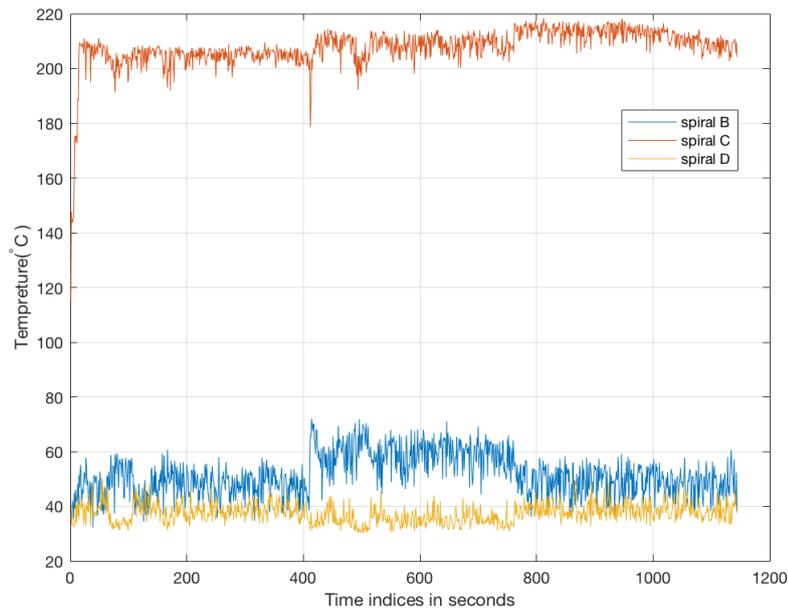


Figure 4.4: An example of 'mouth shape' in experiment one

flow would take more heat. However, since the time indices on the plot are too tight and the measurement results from temperature sensor have too much fluctuation, it is not easy to see the 'mouth shape' in figure 4.8. An example of 'mouth shape' is shown in figure 4.6 and highlighted with the red shadow, this plot takes fewer temperature data which makes the 'mouth shape' easier to observe.

Since this experiment was conducted under high temperature, it was essential to check if the membrane is broken by observing the chip membrane with a microscope, so the lenses of the microscope switched frequently. Nevertheless, when the lenses changed, the temperature of the heater and sensors jumped rapidly as well. Those 'temperature jumps' are pointed out by yellow circles in figure 4.8. Initially, these 'temperature jumps' concluded as the result of convection flow changing. Because there is a hole opened on the protection box, the air was pushed into the box through this hole, when the lenses switched from low power lens to high power lens the lens length would increase convection flow above the combustion chamber. However, this conclusion was overturned after observing the power line of the heater, because when the higher power lens switched to a power lens, both the power and temperature increased immediately as shown in figure 4.7 from Lab-view. Normally if the temperature of the heater goes higher, the resistance goes higher as well, and the current goes down. Since the voltage is unchanged, the power in this case which equals current multiplies with voltage should drop. Therefore these 'temperature jumps' are most possibly some interference signals given by

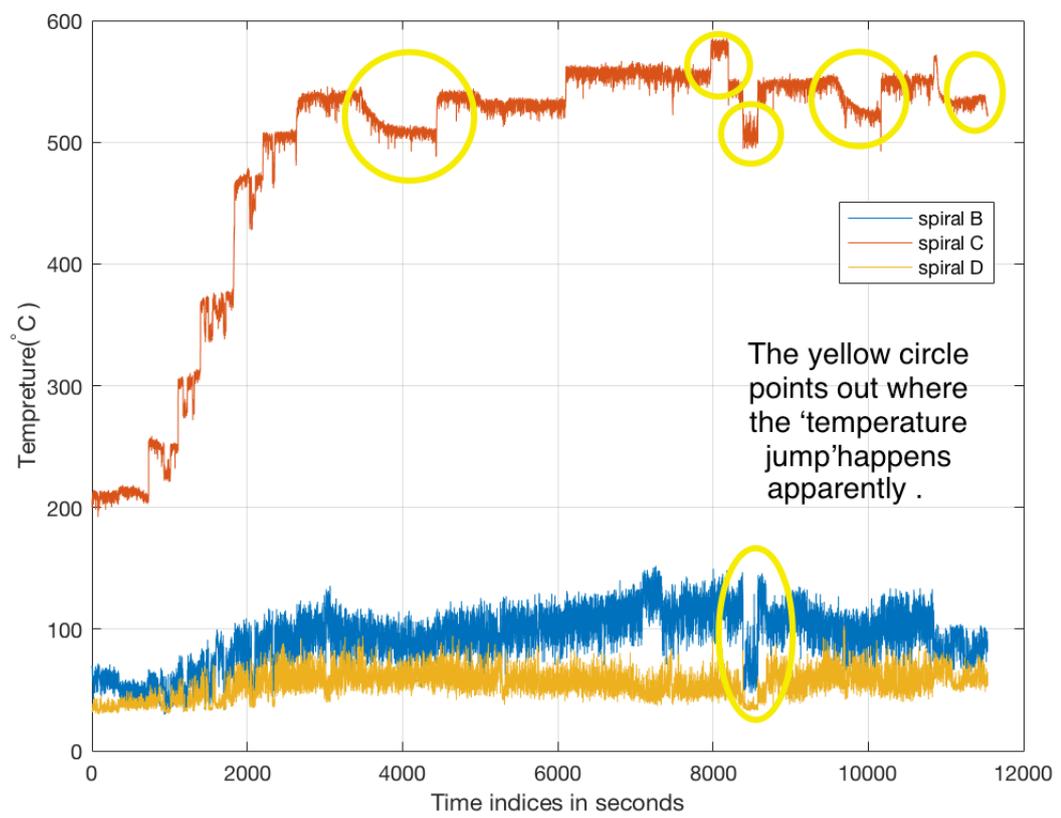


Figure 4.5: The results of experiment two

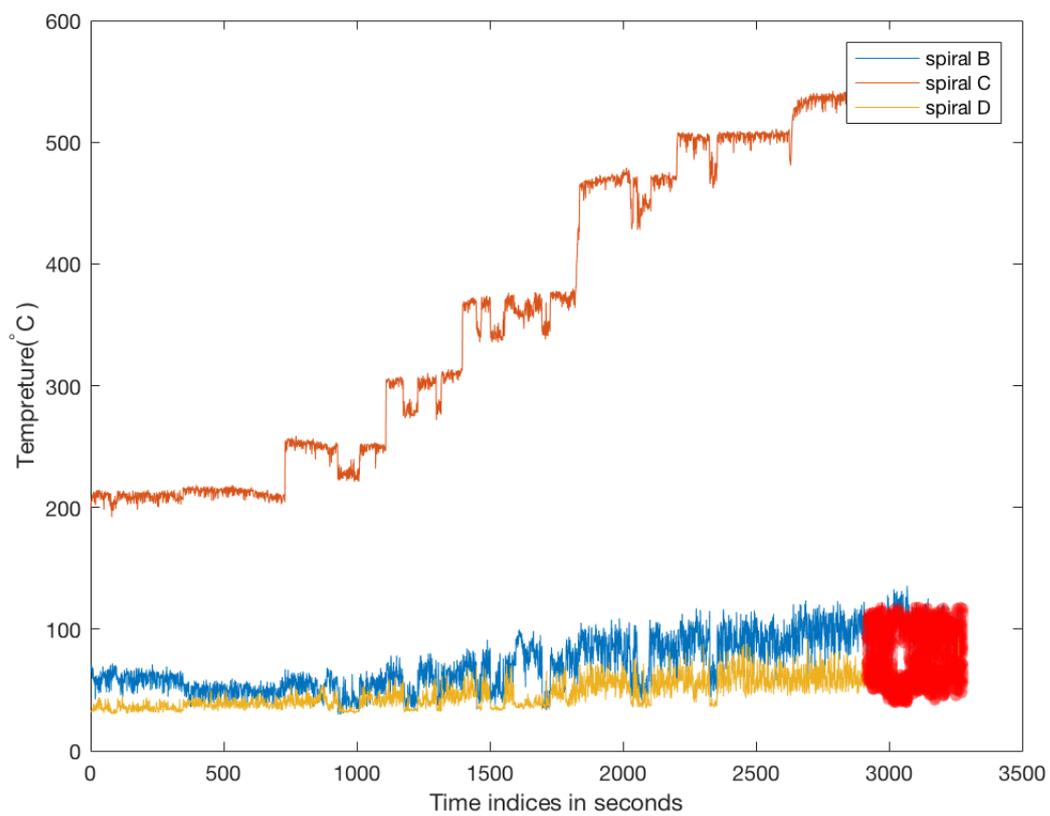


Figure 4.6: An example of the mouth shape of experiment two

the system when switching the lenses of the microscope.

On the other hand, when the heater heated up to 550°C , some 'bulbs' were observed on spiral B². These 'bulbs' indicated that there were some intervals generated between the platinum wire and silicon nitride membrane. If the heater keep on heating up to an even higher temperature, the membrane would take a risk of breaking down due to uneven heating, when the heater temperature was 550°C , the temperatures of spiral B and D are only 80°C and 60°C respectively, which means the temperature of the combustion chamber was way too less than 600°C .

Some conclusions could be drawn from the results of experiment two:

- The heater was not able to heat up the combustion chamber to 600°C .
- Switching the lenses of the microscope will give some interferences on the result
- Adding the only airflow in high temperature would only increase the temperature of spiral B and decrease the temperature of spiral and D. Also, the heater temperature would drop a little bit.

4.4 Experiment three

Experiment three was based on experiment two to investigate when the heater maintained a high temperature, what would happen when gave the methane and air mixture as calculated in the chapter 3.2³.

As observed In Experiment two, when the temperature reached 550°C , the membrane took a risk to break down. To avoid this risk, the heater temperature was always set up to 530°C . This temperature was the average temperature of the spiral, the center temperature was above the ignition point of the methane, so the combustion was expected to happen around the center of the heater.

4.4.1 Experiment before adjustment

This experiment first repeat the same operations as did in experiment two, instead of giving only the air flow, the air-methane gases mixture was given in this case. However, in comparison with the result of experiment two, there was almost no difference, which means there was almost no combustion in this case.

²See picture in appendix A5

³Since the experiment had been done before chapter 3.2 got revised, during the revising process, the mass flow has been changed. Therefore the mass flow values used in the experiment are different from in chapter 3.2.

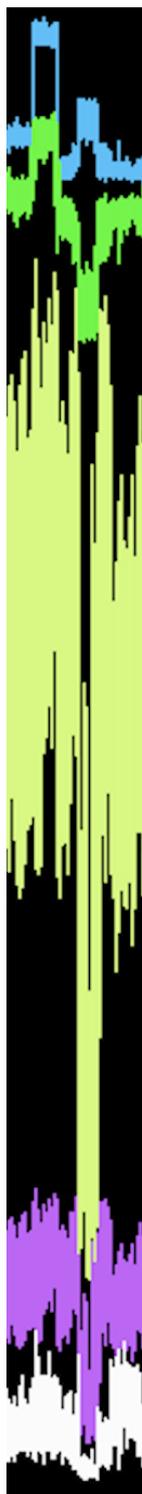


Figure 4.7: Illustration of the influence of switching lenses on heater temperature, the green line is the temperature of spiral C, the yellow line is the temperature of spiral B, both white line and purple line stand for the temperature of spiral D with different scales on temperature.

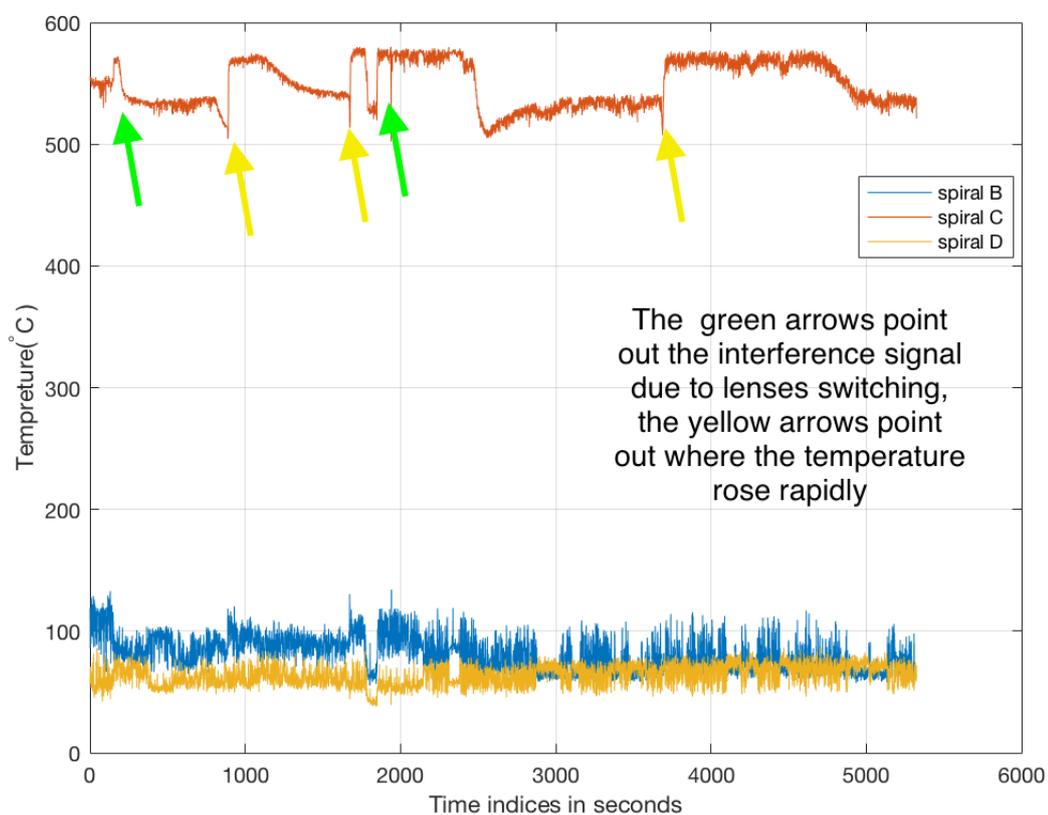


Figure 4.8: The results of experiment three after adjustment

Operation time	Heater temperature	Flow	outlet tube
300s	keep 530°C	only methane flow is given 7.3mg/h	sealed by a tape
500s	keep 530°C	only air flow is given 1000mg/h	open the seal
300s	keep 530°C	only methane flow is given 7.3mg/h	sealed by a tape
600s	keep 530°C	only air flow is given 1000mg/h	open the seal
300s	keep 530°C	only methane flow is given 7.3mg/h	sealed by a tape
1000s	keep 530°C	only air flow is given 1000mg/h	open the seal

Table 4.4: The operation process of experiment three after adjustment

There were two most possible explanations for why there was almost no combustion in this case:

- The amount of methane around the heater center was little, maybe small scale combustion was generated, but the heat was not able to be detected by the temperature difference.
- The gas mixture flow and the flow generated by the vacuum pump would take too much heat away which prevent the combustion.

4.4.2 Experiment after adjustment

In order to make some improvement, some adjustments were made, the operations for the experiment are indicated in the table 4.4:

The result is in figure 4.8. Referring to the figure 4.8 and table 4.4, ignoring the interference signals which are pointed out by green arrows, after giving methane flow in a sealed chamber for few minutes, the massive airflow came in into the chamber and took out a lot of heat which lead to heater temperature decreasing. After a short time, the heater temperature increase to 570°C rapidly, then this temperature kept for a while and gradually went down. This operation was repeated for three times, and the same results were obtained for three times, whenever the temperature reached 570°C, it lasted longer than last time.

Here is one most possible explanation for the result, the combustion chamber was filled with methane. After the massive airflow went in the chamber, the temperature dropped immediately first due to convection. Then the air reacted with the methane around the heater center which would increase the heater temperature. The reason why every time the 570°C lasted longer than last time could because the inlet tube was long enough to accumulate plenty of methane inside, so whenever airflow added, the air flow would mix with the excess methane and this gas mixture started to burn in the center of the heater.

Concerning the results of experiment three, one conclusion draws in this case :

-
- It seems combustion happened after adjustment since there was no additional heat source to heat up the heater to 570°C . However, there was no direct evidence to prove combustion really happened, so the further experimentation is needed.

Discussion

The results from the experiments are quite different from the results expected in chapter 3.2¹, mainly because the heater was not able to heat up the whole chamber to the desired temperature, for the sake of improving this point, three measures could be taken:

1. The temperature of the combustion chamber is decided by the heating power and the thermal resistance, using the material with less thermal conductivity to create the combustion chamber can increase the thermal resistance of the chamber wall, which could make sure higher temperature with identical power.
2. The convection flow from the vacuum pump could also take a lot of heat out of the combustion chamber. Therefore the vacuum pump should be placed away from the chip during the experiment.
3. Using more heaters in the combustion chamber.

On the other hand, the temperature rising still needs to be verified if it is real combustion. The most convenient also efficient way is to release the exhaust gas to a Calcium hydroxide ($Ca(OH)_2$) solution. Because when methane combusts in the air, carbon dioxide is generated, and the Carbon dioxide will react with the Calcium hydroxide and form a white filter immediately.

Finally, in the future, it's necessary to do the same experiments again to verify if the same results are obtained to exclude the random factors which could impact some influences on the outcomes.

¹Since the experiment had been done before chapter 3.2 got revised, The original results are different from the expected results shown in chapter 3.2.

Conclusion and outlook

This report mainly investigates the probability to realize stable combustion in a micro combustion chamber, also the possibility to create a micro combustion chamber with 3D printing aluminum oxide.

In the initial result, it seems a 'likely stable combustion' was realized in microscale combustion. However, it still needs to be verified, some designs of the combustion chamber should be improved in future as well.

For 3D printing combustion chamber with aluminum oxide, the draft design has been sent to one 3D printing company. The possibility is unknown until the reply is obtained.

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