

Design and Construction of a Small Vacuum Furnace

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Abstract. The purpose of this research is designed and constructed of a small vacuum furnace. A cylindrical graphite was chosen as the material of the furnace, the cylinder aluminium and copper sheets were employed to prevent the heat radiation that transfers from the furnace to the chamber wall. A rotary pump used, the pressure of graphite furnace can be pumped up to 30 mTorr and heated up to 700 °C driving by wire and the temperature of the chamber wall is relatively remained too low. In addition, heat loss obtained from the graphite furnace by conduction, convection, and radiation were analyzed. The dominating heat loss was found to be caused by the blackbody radiation, which can thus be used to estimate the relationship between graphite furnace temperature and the drive power needed. The cylindrical graphite furnace has an inner diameter of 44 mm, the outer diameter of 60 mm and 45 mm in height, the 355.5 W of power is needed to drive the furnace to 700 °C.

1. Introduction

In many engineering experiments and scientific research, an environment of high temperature in a vacuum is required for reducing unexpected contaminations and reduces heat convection that causes of heat loss. The application of vacuum system was well recognized in the wide range such as a vacuum packaging, crystal growing [1], annealing [2-3] and the another one is fluxless soldering process [4], silicon layer bonding process [5], and semiconductor layer bonding process [6].

These furnaces are usually operated in the atmosphere, so the sample can be contaminated by dust, steam, and etc. These impurities could be obstructed the interaction between gas and target sample. Therefore, to deal with this problem, the operation in the vacuum chamber is employed.

The purpose of this research is designing and construction of a small vacuum furnace. This system consists of a base plate used the 304 stainless steel a cylinder glasses chamber (closed one side) and the graphite furnace. The aluminium cylinder and copper sheets employed to prevent the heat radiation which transferred from the furnace to the chamber wall.

2. Vacuum furnace design and construction

Figure 1. shown the systems consist of a chamber that made of cylindrical glass, stainless steel plate (base plate), a cylindrical graphite furnace encircled with the ceramic tube, a graphite support made of stainless steel, a ceramic post, and the aluminum sheets.

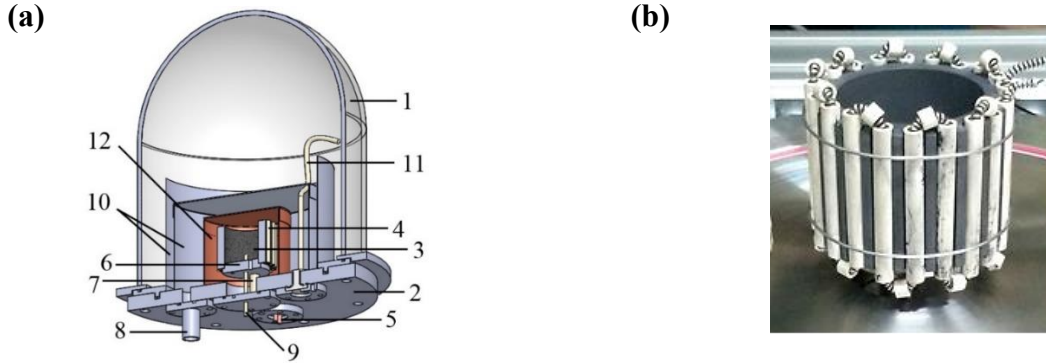


Figure 1. (a) The vacuum furnace consists of (1) close end cylindrical glass, (2) stainless steel base plate, (3) cylindrical graphite furnace, (4) ceramic tube, (5) copper wire in electrical feed-through, (6) graphite support, (7) ceramic post, (8) vacuum pump feed-through, (9) thermocouple feed-through (furnace temperature measured, T_f), (10) the aluminium sheets, (11) thermocouple (chamber temperature measured, T_c), (12) copper sheet (b) the graphite furnace mounted with a ceramic tube for heat conduction, generated by a heating wire inside a ceramic tube.

The base plate was sealed with Viton O-ring, formed a vacuum chamber. The chamber has an inner diameter of 237 mm and height of 270 mm. The type-K thermocouples are used for measurement the furnace temperature (T_f) and the chamber temperature (T_c). A rotary pump was used for evacuated gas in the chamber during operation. The furnace surrounded by the heater wire with ceramic tube insulator as show in Figure 1(b). The graphite was chosen as the material of the furnace because it has been experimentally proven that it nearly perfect absorbs and emitter of radiation [7]. To achieve high isolation, the furnace is mounted on a ceramic post, whose other end is fixed to the base plate.

Center of the base plate is mounted with a thermocouple feed-through for measuring the furnace temperature (T_f). The base plate has four ports surround the center feed-through, two of these are occupied by electrical feed-throughs and thermocouple feed-throughs. The other is for thermocouple wires that used for measuring the chamber temperature (T_c). The third port was for connected to a rotary pump and the fourth port was reserved. The plates and the main equipment of vacuum system are made of stainless steel 304 grade, because of non-rusting occasions and resisting corrosion [8-9].

Outside the furnace, the cylinder aluminum and copper sheets employed to prevent the heat radiation from the furnace to the chamber wall [10], molybdenum and tungsten sheets play a key role in the design of thermal insulation systems in the high temperature furnace. Nevertheless, molybdenum and tungsten are expensive and high melting point, over the range of operating in this works, only 700 °C of temperature which the furnace driven. An aluminium and copper were chosen as the material of the insulation to prevent the heat radiation from a furnace because it has the emissivity coefficient similar to molybdenum and tungsten, but its low cost and the melting point is accordance with the operating range.

3. Theoretical analysis

The analysis of three heat transfer mechanisms was carry on in this section [7]. The thermal resistance of the ceramic post ($\theta_{ceramic}$) was determined the equation (1),

$$\theta_{ceramic} = \frac{L}{kA}, \quad (1)$$

where L is the length of the ceramic post, k is the thermal conductivity and A is the surface area of the ceramic post. Substituting these value into Eq. (1), the ceramic post thermal resistance of 190.7 K/W

was evaluated, which very high value. The heat loss can be calculated using the well-known equation (2),

$$Q = \frac{\Delta T}{R}, \quad (2)$$

where Q is the power, or heat loss, and ΔT is the temperature difference between the inner of graphite furnace and the chamber enclosure. Based on experimental results, when the heating wire is driven at 355.5 W of electrical power and ΔT was 920.25 K. The heat loss due to ceramic post of 4.83 W, only 1.36 % of the drive power was evaluated. At the room temperature and the furnace was driven to 700 °C, the thermal conductivity of 394 W/m K, the thermal resistance of 31-30 K/W. The heat loss of the copper pair of 29.4 W were evaluated which was 8.25% of the drive power.

The convection loss under a typical room temperature ambient was evaluated by equation (3),

$$Q = hA\Delta T = (1/\theta_{con})\Delta T, \quad (3)$$

where $\theta_{con} \equiv 1/hA$ is the equivalent convection thermal resistance from the platform to the ambient, h is the natural convection heat transfer coefficient and A is the surface area of the graphite platform. For this platform with the atmosphere condition the convection heat loss of 27.61 W will be calculated, it was 7.77 % of the drive power value and therefor not considered important. The heat loss due to convection of 0.02 W was calculated with the pressure of 1 Torr. It was very small value.

Since the graphite is very close to an ideal blackbody, we can employ the blackbody radiation theory for power radiation determination. The Stefan-Boltzmann formula [11] gives the net power radiated per surface area of the blackbody with the equation (4),

$$P_{rad}(T) = \sigma A e (T^4 - T_A^4), \quad (4)$$

where $\sigma = 5.6710^{-8}$ W/m² K⁴ is the well-known Stefan constant, A is the surface area, e is the emissivity, T is the blackbody temperature in Kelvin, and T_A is the ambient temperature. Applying equation (4) to the graphite furnace, P_{rad} becomes the heat loss due to radiation.

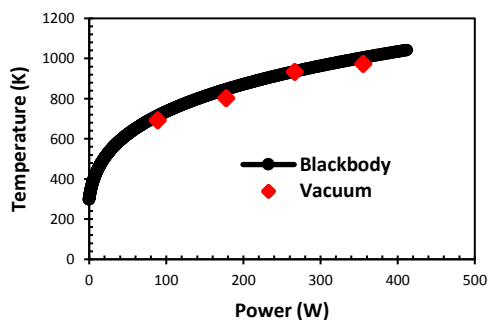


Figure 2. Shown the graphite furnace temperature calculated based on blackbody radiation theory, in atmosphere condition at 298 K and temperature in vacuum condition.

If the conduction and losses are ignored, $P_{rad}(T)$ is the power that has to be supplied to the platform continuously in order to keep the platform at a temperature of T . Figure 2. displays the calculated temperature versus drive power. From the curve and the heat loss that we calculated above, we see that, the radiation heat loss is much larger than the conduction heat loss or the convection heat loss.

4. Experimental and measurement results

It takes less than 1 min for the pressure to decrease from 760 Torr to 1 Torr. After pumping for 2 hours, 30 mTorr is achieved as shown in figure 3(a), which is the lowest pressure obtained with the configuration. The chamber was pumped down and kept constant at 30 mTorr then the furnace temperature (T_f) and was measured temperature inside the chamber (T_c). The furnace driven with laboratory made 77 VDC power supply. The furnace power can be verified by PWM method used the Arduino microcontroller board, the temperatures were collected and real-time monitor with serial chart program in the computer. Where $R=10\ \Omega$ is the resistance of the heating wire, the power 355.5 W was used to drive the platform to 700 °C. At this furnace temperature, the environment temperature in the chamber is only 53 °C as shown in figure 3(b).

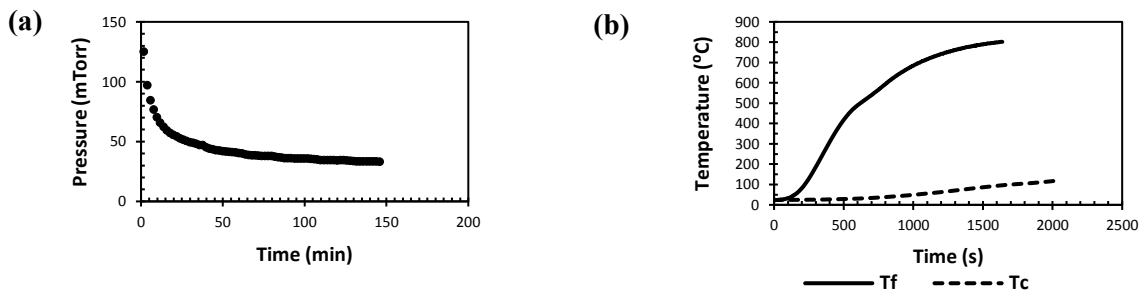


Figure 3. (a) The pressure versus a time to reached the target pressure. (b) Temperatures inside the graphite furnace and the temperature inside the chamber versus a time to reach the target temperatures.

During operation and measurements, a fan was used to blow only at under the base plate of the chamber. The furnace was not driven beyond 700 °C because it is the highest temperature we would ever need in present research, driving to higher temperature might shorten the life of the heating wire.

The next part we would show the result that derived by 15%, 30%, 45% and 60% of a maximum power. The Arduino board was used to control the power that supplied to the graphite furnace. The maximum power is 592.5 W. Thus, 88.875 W, 177.75 W, 266.625 W and 355.5 W is the level of power calculated by the percentage of a maximum power. The temperatures inside graphite furnace and the temperature inside the chamber versus a time to reach the target temperatures were represented in figure 4.

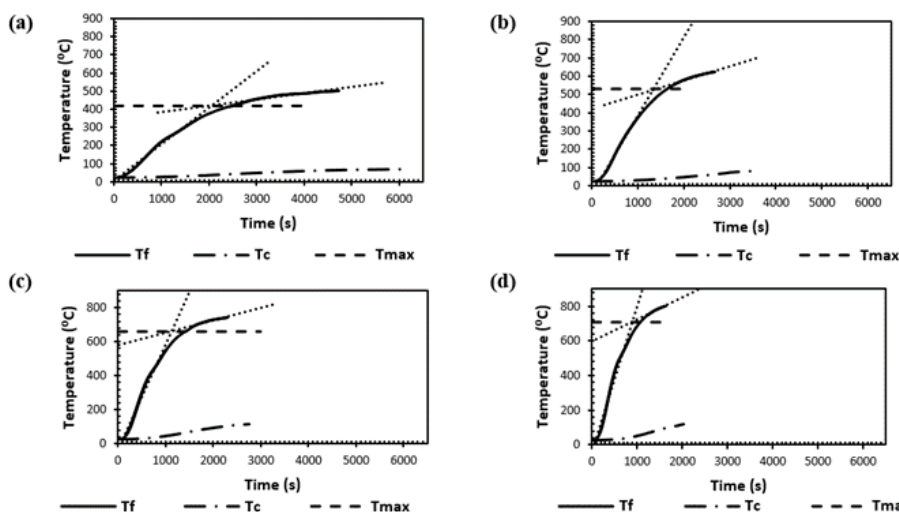


Figure 4. The result derived from percentage of a maximum power (a) 15% (b) 30% (c) 45% and (d) 60%.

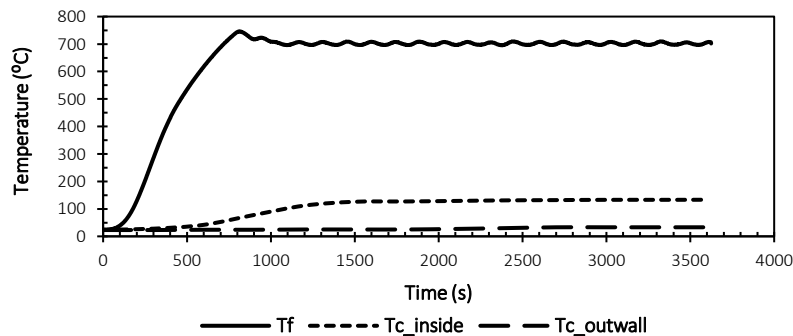


Figure 5. The appearance temperature of the furnace when setting point was 700 °C with a laboratory made PID controller.

Figure 2. Also shown the graphite furnace temperature measured in vacuum, 15%, 30%, 45% and 60% of a maximum power were applied to find the power which can reach the target temperature and the blackbody radiation curve presented in figure 2. From these two curves, we see that, for the same drive power, the graphite furnace temperature measured in a vacuum is nearly to the blackbody radiation value. This result agrees with the fact that convection heat loss is negligible in a vacuum environment. We also see that the blackbody temperature is just a little bit higher than the platform temperature measured in vacuum. The graphite furnace in a vacuum has less heat loss and this measurement result shown that the graphite is indeed a nearly ideal blackbody. It absorbs and stores the heat produced by the heating wire and in turn radiates heat from the surface.

Finally, the stability of the furnace was demonstrated with the target furnace temperature was set at 700 °C. The average temperature of the furnace was 701.95 °C with an oscillation in the range of 698-705 °C as shown in figure 5. The maximum temperature in a vacuum chamber was 129 °C and the maximum temperature of vacuum chamber wall was 35 °C. The results shown that the high target furnace temperature was set with the lowly vacuum chamber wall.

5. Conclusion

We found the target temperature was reached by use the 60% power of the maximum power in short time (about 17 min). The maximum temperature inside the chamber is 129 °C, occur in overshoot zone. Nevertheless, each result in four conditions was proved the temperature in a chamber is relative cool while the furnace climb to the target temperature. Using the small and efficient vacuum furnace constructed, we have developed the small vacuum furnace for the science and engineering research which require the environment of high temperature in a vacuum. The furnace is very convenient and economical to use. It provides a clean environment to develop new processes.

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