CHAPTER 4

DESIGN, FABRICATION AND PERFORMANCE STUDY OF EXPERIMENTAL MICROWAVE SINTERING SETUP

4.1 Selection of microwave oven

It is very important to select a microwave furnace that fulfils the requirement of this study. The microwave furnace must be able to control and display the time and temperature as it is the controllable factors in this study. Looking at the type of microwave furnace in the market, generally there are of two types that we can put into consideration. One is the industrial microwave oven and another type is the domestic microwave oven.

The industrial microwave oven employs a high energy power source with long life. The system also has a unique internal protection feature (patented) that traps stray microwaves, thus making the system completely safe to operate. A new exclusive type of forced-air system used in conjunction with microwave energy (patented) accelerates the removal of moisture quickly and efficiently. In addition, a straightthrough conveyor assures uniform exposure to the microwaves. Because of its high processing capacity, the sintering process can take a shorter time (sintering process of the same condition can be done simultaneously).

The domestic microwave type ovens available to date employ a relatively low power source as compared to the industrial power source. In addition, the typical

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domestic microwave oven is considerably less efficient, up to 25% less, in converting electrical energy into microwave energy. The domestic microwave oven has the time controller but lacks the temperature controller because temperature is not important to serve its purpose as cooking and heating food.

After considering the advantages and disadvantages of both types of microwave furnace, it is obvious that the industrial microwave oven is a better option. However, it costs much higher than the domestic microwave oven. So, a domestic microwave oven was chosen as the sintering furnace and a 1kW home use microwave oven (Samsung, model M197DN) with 2.45 GHz single mode cavity was purchased as shown in Figure 4.1.



Figure 4.1 Microwave oven selected

The microwave oven purchased was modified in collaboration with G.B Kiat (Microwave Sintering of Tin Base Alloys and Composites, 2005) for this study.

4.2 General design of the modification of microwave oven

The first step of modification of the microwave oven is to analyze what is lacking in the system but needed in the experiment. It is obvious that the microwave purchased does not have temperature controller and display. Therefore, a thermocouple was needed to measure the temperature of the specimen.

A Chromel-Alumel thermocouple type K with a diameter of 1mm was connected in series to a Fuji temperature controller cum indicator, model PXZ-4. This thermocouple was inserted in an alumina protection tube which is installed in the cavity of the microwave oven and through the top of the microwave oven. A schematic diagram of the system is shown in Figure 4.2.





(2) Thermocouple; (3) Microwave oven; (4) Temperature controller; (5) Power source

For the cavity of the microwave oven, modification must be done to house the specimen. This is because the specimen cannot be exposed directly to the microwave energy as the metallic product will reflect microwave energy. Therefore, a fibre cube

was used to house the specimen. The thermocouple must be in contact with the specimen in the fibre cube through a hole of the cover, cut out from the fibre cube.

At the bottom of the microwave cavity, a fible blanket was installed. The fibre cube was located at a position such that there is no gap between the fibre blanket and the end of the alumina protection tube as shown in Figure 4.2.1



Figure 4.2.1 Schematic diagram of the microwave oven and the position of the fibre blanket, fibre cube and alumina tube.

4.3 Installation of fibre blanket

Inside the microwave oven is a rotating plate where the food is placed for heating. The rotating plate gives a uniform heating to the food heated or cooked. However, in this study rotation is not necessary as the size of the sample is far too small to consider the need for uniform heating (cylindrical bearing of approximately 20mm in diameter). In addition, by eliminating the rotating plate, the ease of measuring the sample's temperature was increased.

The rotating plate can be taken out from the oven cavity directly. However, the plate is actually driven by a rotary head and it is planted at the base of the microwave oven. This rotary head cannot be taken out and will still rotate whenever the microwave oven operates. Simultaneously, it is necessary also to consider the insulation of the microwave oven. Insulation is essential because the sample which is a metallic product cannot be directly sintered by microwave. Metallic product will reflect microwave energy.

To solve the two problems, a fibre blanket was installed as shown in Figure 4.3. The fibre blanket is 130mm X 130mm in size and a thickness of 60mm. A hole of 50mm diameter is cut through the centre of the fibre blanket to accommodate the rotary head. The dimensions are shown in Figure 4.3.1. Therefore the movement of the rotary head will not affect the experimental procedure.







Figure 4.3.1 The fibre blanket dimensions

4.4 Installation of alumina protection tube and thermocouple

As mentioned, the domestic microwave oven does not possess a means to monitor and control the temperature. Therefore, a thermocouple must be inserted in the system and connected to a temperature controller. The thermocouple is of type K, chromel-alumel which is the 90/10 Ni/Cr alloy, commonly used in thermocouples, in conjunction with a 95/5 Ni/Al alloy. Similar to heating elements, it has a maximum operating temperature of 1100°C. The thermocouple must be in contact with the specimen to measure and monitor its temperature.

This thermocouple is protected by an alumina protection tube. Alumina or aluminium oxide (Al_2O_3) is chosen because of its properties. Its high free energy of formation makes alumina chemically stable and refractory, and hence it finds uses in containment of aggressive and high temperature environments as in this experiment. The high dielectric constant of alumina coupled with low dielectric loss particularly at high frequencies leads to a number of microwave applications including windows for high power devices and waveguides and thus suitable for this particular study.

Figure 4.4 shows the thermocouple and Figure 4.4.1 shows how it is inserted into the alumina protection tube. The alumina protection tube is 10mm in diameter with length of 180mm. It is fixed inside the microwave oven at the centre and through the top of the microwave oven. A hole of 10mm is drilled on the top of the microwave oven to accommodate the alumina protection tube. The alumina protection tube is inserted into the hole at a position such that the distance between the end of the thermocouple inside the oven and the top of the oven is125mm. Then the gap between the alumina protection tube and the top of the oven is sealed using silicon. Silicon can stand up to 500°C and thus used to seal the gap to prevent microwave leakage. Microwave leakage can lead to insufficient heating and may become hazardous to the near by user.



Figure 4.4 Thermocouple

Figure 4.4.1 Thermocouple inside the alumina protection tube

4.5 The making of fibre cube

A fibre cube was placed between the fibre blanket and the end of the alumina protection tube as shown in Figure 4.5. This fibre cube is the medium that is used to contain the specimen in the experiment.



Figure 4.5 Fibre cube to house the specimen during experiment

The fibre cube acts as an insulation to avoid heat dissipation during sintering. It also plays a role to prevent direct exposure of the specimen to microwave energy as metallic product reflects microwave energy. The cube is of 78 X 78 X 50mm in dimension and it can resist 500°C of temperature.

With the presence of the insulation package, the system does not require susceptor or secondary coupler. A susceptor or secondary coupler is usually used to keep temperature of sample more uniform and provide the initial heating of specimen before it starts coupling with microwave.

To put in the sample in the fibre cube, a cavity of 21 X 21mm is cut out at the centre of the cube as shown in Figure 4.5.1. The dimensions are design in such a way so that it can best fit the cylindrical bearing of 20mm in diameter which is the size of the specimens throughout the study. Therefore, there will be minimum space inside the cavity. The depth of cut is 20mm and a part of the fibre being cut is used as a cover to close the cavity. On the cover, a hole of 4mm in diameter is cut at the centre

so that the thermocouple can go through it to touch the surface of the specimen as

shown in Figure 4.5.2 and Figure 4.5.3.



Figure 4.5.1 Dimension of fibre cube cut out to accommodate the specimen







thermocouple is in contact with the specimen.

Figure 4.5.3 The fibre cube. Notice that there is a hole on the cover.

4.6 Modification of electronic circuit and installation of temperature controller

When the thermocouple is added to the system, it means that a modification of the electronic circuit must be done too. This is shown in Figure 4.6.



Figure 4.6 Modified electronic circuit of microwave oven.

Ideally, there must be constant dwell time and constant temperature during sintering. However, this is not possible in this system because of the mechanism operation of microwave oven. The operation of the microwave oven or more precisely, the operation of magnetron is used as a mechanism to control the temperature (temperature controller). The temperature will not be constant but instead will fluctuate around the preset value. When the temperature is more than the preset value, the temperature controller will cut off the operation of magnetron. As a result, the temperature of the microwave oven will decrease. When the temperature is below the preset value, the temperature controller will restore the magnetrons operation.

A solid state relay is used to give on and off command to the magnetron. Solid state relay or semiconductor relay (also called SSR) is a semiconductor device that can be used in place of mechanical relay to switch electricity to a load in many applications. It is purely an electronic device, normally composed of a low current control side and a high current load side (switching side). The solid state relay and the temperature controller cum indicator is connected and installed in a control cubical for safety purposes and easy manipulation as shown in Figure 4.6.1.



Figure 4.6.1 Control cubical of the temperature controller

An additional four inches cooling fan is installed bas shown in Figure 4.6.2. This is to prevent the microwave oven from overheating. This particular study needs to reach a sintering temperature as high as 220°C and as along as one hour. This temperature might exceed the range of conventional purpose of a domestic microwave oven that is to cook and heat food. The thermostat of the microwave oven will sense rising temperature and the microwave oven will terminate its operation immediately for safety measure. This additional fan will assist the sintering process by cooling the microwave oven.



Figure 4.6.2 An additional fan installed outside the microwave oven

4.7 Testing of overall system

After modification, it is wise to start the experiment by testing the system first. This is to ensure its feasibility and functionality of the system fulfils the requirement of the study. If there is even a minor error, it might lead to system failure and thus the

experiment cannot be conducted. Microwave leakage can raise potential danger to the user.

In the testing, a specimen of equal composition as in the experiment is used to sinter. The sintering temperature is set to the highest condition that is 220°C and the time is set to 1 hour. During the testing it is important that any sign of danger or possible system breakdown noted so that the operation can be stopped immediately to avoid more harm to be imposed on the system. The procedure of the testing is as follows:

- a) A specimen is placed inside the fibre cube. Carefully put the cube between the fibre blanket and the alumina protection tube after closing its cover.
- b) Make sure that the hole on the cover is around the centre of the alumina protection tube and insert the thermocouple in the alumina protection tube.
- c) The tip of the thermocouple must be in contact with the specimen inside the fibre cube. Close the microwave oven door.
- d) Set the time to more than one hour (around 62 minutes) to accommodate the dwell time. On the microwave oven and start sintering.
- e) The temperature controller can only be set when the microwave oven has started the sintering process. Set the temperature to 220°C.
- f) The temperature controller will either display the preset temperature or the current temperature. Select the display current temperature mode.
- g) When the temperature reached 220°C, use a stopwatch to set the time.
- h) Monitor the whole sintering process to observe if there is any peculiarity of the system. If there is any sign of danger like smoke coming out stop the system immediately.

- i) When time is up, stop the system manually.
- j) The specimen is taken out. Wait for the oven to cool down and repeat testing.

At first, the system stopped which shows the system is unstable. After repeated testing, it is concluded that the system is working and the modification is a success. However, if the microwave is used to sinter one specimen after another with only a few minutes of lag, the system seem to take more time to reach the desired temperature. On the other hand, the more stable the system becomes. To prevent the system from becoming unstable, a few safety measures should be followed.

The safety steps suggested are:

- a) The fibre cube must be handled with care because of its brittle nature.
- b) There should not be a gap between end of the alumina protection tube and the fibre cube or at least it must be minimized to avoid the specimen from reflecting the microwave energy and thus results in system shutdown.
- c) The fibre blanket can tear apart easily. Therefore, handle with care to avoid thinning of the fibre blanket.
- d) Thermocouple must be in contact with the specimen for accurate measurement of the temperature.
- e) The preset temperature must be less than the sintering temperature to avoid base metal from melting (highest condition is 220°C and tin melting point is 232°C) because of fluctuation of temperature by the system.

4.8 Temperature fluctuation

Even though it is essential that the sintering temperature be constant for this study, it is impossible because of the nature of the microwave heating mechanism itself. Microwave heating can produce hot spots or "thermal runaway." This occurs when one section of the material absorbs the microwave energy preferentially over the bulk material.

Heating is a diffusion process whose rate is limited by the coefficient of thermal diffusivity. If for any reason, local diffusion rate is much less than microwave power dissipation rate, local temperature will increase rapidly. With increasing temperature, the properties of the material change. If the changes lead to acceleration of microwave power dissipation at this local point, temperature will increase more rapidly. Result of such a positive feedback is the formation of a hot spot which is a local thermal runaway.

As a result, thermal runaway destabilizes the temperature of specimen and subsequently causes fluctuation of temperature. Range of fluctuation temperature is even intensified by the on-off mechanism of magnetron commanded by temperature controller.

For simplicity, the time and temperature observed during the testing is presented in the graphs below. Figure 4.8 shows the temperatures of the first minute of sintering. The preset temperature is 210° and 60 minutes is the duration of sintering. Figure 4.8.1 shows the time and the corresponding temperatures of the entire sintering process.



Figure 4.8 Temperatures of the first minute of sintering.





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From the testing of the system, it is found that the dwell time is around 30 seconds. The temperature usually starts at room temperature and it took less than a minute to reach to the desired temperature, in this case 220°C. After 7 minutes, the temperature can reach as high as 232°C and as low as 196°C. This shows that the fluctuation range is quite large. The specimen is taken out after sintering and it is left to cool down at room temperature. The cooling time is around 5 minutes.