
CHAPTER 3

METHODOLOGY

3.1 Selection of material design

The metals used in this study are tin, copper and antimony. Tin is a silvery-white luster metal, with a bluish tinge. It is malleable to a certain extent and is ductile and possesses a highly crystalline structure. Its hardness is only slightly greater than that of lead, and its electrical conductivity is approximately one seventh that of silver. Tin takes on a high polish and is resistant to atmospheric corrosion. It is resistant to distilled, sea and soft tap water. However, tin is attacked by strong acids, alkalis and strong salts. The presence of oxygen within solution accelerates the attack. One of the most important properties of tin is the ease with which it alloys or mixes with the majority of other metals.

Copper has a face centered cubic crystal structure. It is yellowish red in physical appearance and takes on a bright metallic luster when polished. It is tough, ductile and malleable. Copper is the best conductor of electricity next to silver, having a conductivity 97% that of silver.

Copper is generally corrosion-resistant to rural, marine and industrial atmospheres, it is resistant to various waters, saline solutions, soils, non-oxidizing mineral and organic acids and caustic solutions. However, copper is attacked by

oxidizing acids (e.g. nitric), moist ammonia, and halogens, sulphides and solutions containing ammonia ions.

Although antimony is not a common element, it is found in over 100 mineral species. While it can be found in elemental form, it is more often found as a sulfide (Sb_2S_3) in stibnites, as a antimonide of heavy metals or an oxide.

Metallic antimony appears a bluish white and possesses a metallic luster. It has a flaky, crystalline structure and brittle in nature. It is unaffected by air under ambient conditions and burns brilliantly when heated, producing Sb_2O_3 fumes. Metallic antimony is a poor conductor of heat and electricity. It has a hardness of 3 to 3.5.

Table 3.1 shows the composition of all three materials used in this study and Table 3.1.1 shows the density and melting point of the respective materials.

Material	Composition (%)
Tin (Sn)	70
Copper (Cu)	20
Antimony (Sb)	10

Table 3.1 The composition used in this study.

	Density of solid, 20°C (gm/cm^3)	Melting Point (°C)
Tin (Sn)	7.29	231.9681
Copper (Cu)	8.93	1084.87
Antimony (Sb)	6.69	630.755

Table 3.1.1 Density and melting point of tin, copper and antimony (Data taken from J.P Schaffer (1999), The science and Design of Engineering Materials)

3.2 Fixed Parameters

Fixed parameters are parameters that have constant value throughout the experiment conducted. For simplicity, the parameters are listed in Table 3.2 along with its value.

Fixed Parameter	Value
Weighing	+ 0.05 gram
Mixing time	6 hours
Compaction holding time	4 minutes
Cooling temperature for conventional sintering	Room temperature (32°C)
Boiling time (for porosity test)	30 minutes
Vacuum time (for porosity test)	30 minutes

Table 3.2 Fixed parameter and its value

3.3 Controllable Variable Parameters

Controllable variable parameters are factors which can easily be controlled. There are four controllable variable parameters in this study. They are sintering temperature, sintering time, sintering atmosphere and compaction pressure. Sintering time, sintering temperature and sintering atmosphere are also the most important factors involved during sintering process. The influence of these factors on the experiment is described below.

3.3.1 Sintering temperature

Increasing the sintering temperature greatly increases the rate and magnitude of any changes occurring during sintering.

3.3.2 Sintering time

Although the degree of sintering increases with increasing time, the effect is small in comparison to the temperature dependence. The loss of driving force with increasing time at any temperature is one of the reasons why it is very difficult to remove all porosity by sintering.

3.3.3 Sintering atmosphere

Nearly all metals of technical importance react with the gas of their surrounding atmosphere even at room temperature, but more so when treated at higher temperatures. The most important reason for using special sintering atmospheres is to provide protection against oxidation and re-oxidation of the sintered metal compacts. There are many other ways in which a sintering atmosphere can influence the basic sintering process. By reducing the oxides the atmosphere may create highly mobile metal atoms. Gas atoms of the sintering atmosphere can enter the sintering compact via interconnected pores. They may later get trapped in closed pores, thus hindering their shrinkage. Gas atoms of the sintering atmosphere may also diffuse into the metal. At times these atoms might also alloy with the metal.

3.3.4 Compaction pressure

Compaction pressure of the material does not affect the sintering process.

However, it will affect the properties of the material after it underwent the sintering process.

3.4 Uncontrollable Variable Parameters

Uncontrollable variable parameters or noise factors are factors that are difficult or impossible or too expensive to control. There are three types of noise factors: outer noise, inner noise, and between product noise. Noise factors are primarily response for causing a specimen's performance to deviate from its target value. Hence, parameter design seeks to identify settings of the control factors which make the specimen insensitive to variations in the noise factors, i.e., make the specimen more robust, without actually eliminating the causes of variation.

The uncontrollable variable parameters identified in this study are temperature fluctuation in microwave sintering, oxidation of the specimen in conventional sintering and thickness variation of the specimen during compaction. Figure 2.1 is modified in relation with the controllable and uncontrollable variable parameters discussed above and shown in Figure 3.4.

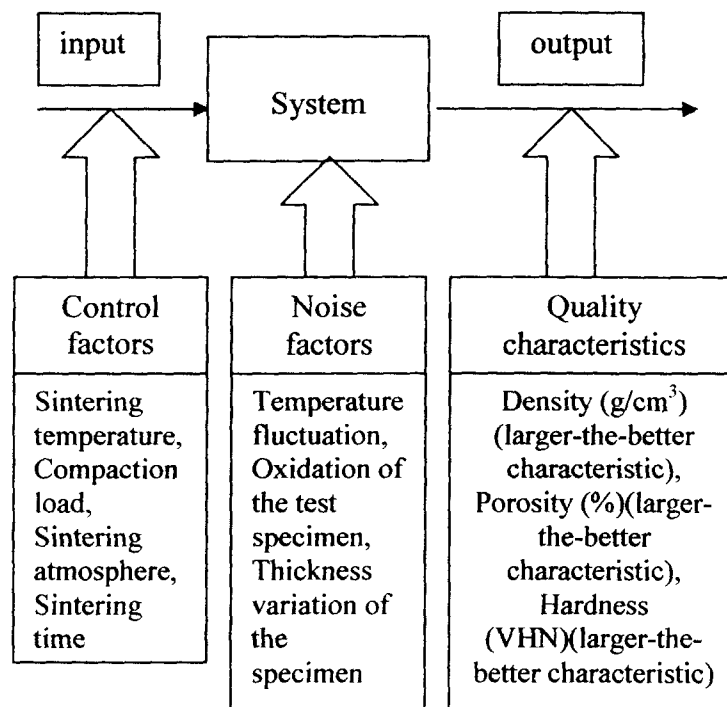


Figure 3.4 Factor-characteristic relation diagram

3.5 Experimental Condition

In this study, Taguchi Parametric Robust Design, based on orthogonal array (L_8) has been used to plan the experimental conditions. The controllable factors that are chosen are sintering temperature (A), compaction pressure (B), type of heating (C) and sintering time (D). The uncontrollable or noise factors are identified as temperature fluctuation, oxidation of the specimen and thickness variation of the specimen.

Each parameter has two level values. The high and lows of the control factors are given in Table 3.5.

Parameter	Low Level (0)	High Level (1)
A: Sintering temperature (°C)	140	220
B: Compaction pressure (MPa)	156	312
C: Type of heating	microwave	conventional (Ar gas)
D: Sintering time (min)	30	60

Table 3.5 Level settings of controllable factors

The higher and lower levels of these factors are designated by numerical symbols 1 and 0 respectively in Table 3.5.1

Experiment number	Column number							Condition
	1	2	3	4	5	6	7	
1	0	0	0	0	0	0	0	$A_0B_0C_0D_0$
2	0	0	0	1	1	1	1	$A_0B_0C_1D_1$
3	0	1	1	0	0	1	1	$A_0B_1C_0D_1$
4	0	1	1	1	1	0	0	$A_0B_1C_1D_0$
5	1	0	1	0	1	0	1	$A_1B_0C_0D_0$
6	1	0	1	1	0	1	0	$A_1B_0C_1D_1$
7	1	1	0	0	1	1	0	$A_1B_1C_0D_1$
8	1	1	0	1	0	0	1	$A_1B_1C_1D_0$
Basic mark	a	b	ab	c	ac	bc	abc	
Assignment of factors	A	B	A x B	C	AxC	D	A x D	

Table 3.5.1 Planning of experimental conditions based on $L_8(2^7)$ orthogonal array design.

As only four control factors are under study and three interactions effect are assumed, an internal orthogonal array with seven columns is found to be suitable and hence $L_8(2^7)$ is chosen for planning the experimental runs. Three replications are used under each set of experimental conditions to eliminate the effect of noise factors. This

design is shown in Table 3.5.1. Actual experimental conditions in different runs are reproduced in Table 3.5.2 from Table 3.5 and Table 3.5.1.

Experiment No	Sintering Temperature (°C)	Compaction Pressure (MPa)	Type of heating	Sintering time (min)
1	140	156	microwave	30
2	140	156	conventional	60
3	140	312	microwave	60
4	140	312	conventional	30
5	220	156	microwave	30
6	220	156	conventional	60
7	220	312	microwave	60
8	220	312	conventional	30

Table 3.5.2 Actual experimental conditions in different runs

3.6 Experimental Procedure

3.6.1 Powder preparation

There are 8 experimental runs with each experimental condition repeated 3 times. Therefore a total of 24 specimens were prepared. Every specimen was of the same weight (10g) and also the same composition as shown in Table 3.6.1

Material	Composition, %wt
Tin (71 μ m)	70
Copper (150 μ m)	20
Antimony (150 μ m)	10

Table 3.6.1 Composition of material

3.6.2 Powder Mixing

The term 'blending' is strictly applied to one component operation, whereas mixing involves more than one type of powder. In this study, tin powder of 71 μ m was mixed in Pascal ball mill with copper powder (150 μ m) and acicular antimony powder (150 μ m). The aim was to mix the powders only as long as necessary to achieve a through mix and to fix a uniform apparent density of the mix from batch to batch. The apparent density of the mix tends to increase with mixing time. The equipment used to mix is shown in Figure 3.6.2.

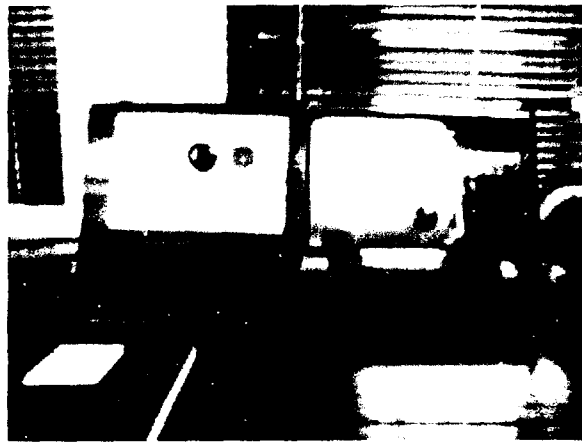


Figure 3.6.2 Mixer

This mixer can support 4 bottles of mixed powder per session. In this study, every bottle contained a 21g of tin powder, 6g of copper and 3g of antimony. So, to make 24 specimens with every specimen weight approximately 10g each, two batches were run. The number of balls inserted in each bottle was determined by the formula of

$$\text{Number of balls} = \frac{\text{Weight in grams} \times 2}{3.4} \quad \text{----- Equation (1)}$$

Therefore, for each bottle, 18 balls were inserted. The mixer ran for 6 hours with the speed of 350 m/s. The apparent density of the mix tends to increase with mixing time.

3.6.3 Powder Compaction

For powder compaction, split die systems was used in this study. This system required two die holding plates to carry the upper die and lower die. Each plate was controlled and moved independently. Compaction presses for powders are of two types- mechanical or hydraulic. Hydraulic presses produce working force through the application of fluid pressure on piston by means of pumps, valves, intensifiers and accumulators. Inherent in the hydraulic method of drive transmission is the capability to provide infinite adjustment of stroke speed, length and pressure within the limits of press capacity. Also, full tonnage can be extended throughout the complete length of the stroke. In this study, a manually operated hydraulic press is used as shown in Figure 3.6.3.



Figure 3.6.3 Manually Operated Hydraulic Press

To begin compacting, the following steps were followed:

1. Raise the front safety guard and place the 10g of mixed powder on the lower bolster pressing face covering the piston. The work is positioned centrally and lowers the safety guard. The top bolster pressing face attached to the lead screw, is screwed down by turning the lead screw handle clockwise.
2. Rotate the pressure release handle clockwise until it has tightened firmly. This closes the pumping system so that it is ready to build up the pressure with the pump handle grip
3. Start to pump the press by pulling and pushing gently, but smoothly, on the pump handle grip. (The correct leverage and force is applied by holding at the rubber handle.)
4. It will take a few strokes to build up pressure in the oil, but once resistance is offered by the work to the raising of the piston, the load will be indicated on the load gauge. Keep on pumping until the required load is achieved.
5. If the work being pressed is compressing to the extent that the piston is traveling its maximum distance, release the pressure by turning the pressure release handle anticlockwise by about one complete rotation. This will allow the piston to return to rest. The work will sink down with the piston and a gap will be created between the work and the top bolster pressing face.
6. Turn the lead screw handle clockwise to bring the top bolster pressing face onto the work again, and reapply the load from the piston (tighten pressure release handle and pump via pump handle).
7. When the required load is indicated on the load gauge stop pumping. Hold the pressure for as long as required. There might be a slight decrease in the pressure applied, indicated at the gauge, as the work being pressed may relax

over time. Bring the pressure back up to the required load by pulling gently on the pump handle grip.

8. To release the load on the work turn the pressure release handle anticlockwise by about one complete rotation. Take out the green compact. Figure 3.6.3.1 shows the top and lower bolsters pressing face, die and piston.



Figure 3.6.3.1 The top and lower bolster pressing face, die and piston

3.6.4 Sintering

The compact or specimen is given its strength by sintering. In this study, the specimen will either be sintered conventionally in an Argon gas environment or sintered in a microwave furnace, depending on the experiment condition.

a. Conventional Sintering

To start sintering the specimen, the steps below are followed:

1. Place the specimen in a ceramic boat and insert it into tube furnace. It is best to place the specimen at the middle of the tube furnace. The ceramic boat can occupy three specimens at a time.
2. Open the Argon gas tank. Make sure that the furnace is filled with Argon gas before operating the controller by looking at the bubbles

coming out from the tube that is connected to the end of the tube furnace.

3. Switch on the controller and the display will show the measured temperature and set point. Hold to the Run/Hold button to go to the Reset mode.
4. Then press Page until ProG LiSt is displayed to start a new program. Next, Scroll button is pressed to display segment number, type or to access the parameters appropriate to the type of segment chosen. The Down or Up button is used to move to the segment to be created, change the required segment type or alter the values.
5. For the first segment, the rmPr (ramp rate) segment type is chosen. The tGt (target set point) for this segment is either 140°C or 220°C. The rAtE (ramp rate or rate of temperature change) is set at 5°C per minute
6. For the second segment, the dwEll (dwell) segment type is used. The dur (duration) of this segment is the time in minutes to remain at the previous target temperature. Either 30 or 60 minutes are chosen for this study.
7. The last segment type will be End which means that the tube furnace will stop heating the specimen and it will cool down on its own.

b. Microwave Sintering

For this study, a domestic microwave oven was modified to sinter the specimens. A more detailed description of the modification is given in

Chapter 4. To sinter the specimen in a microwave oven, the steps below were followed:

1. Place the specimen in the fibre cube and put the cover on top of the specimen.
2. Put the fibre cube on the hollow fibre blanket. Assure that there is no gap between the alumina protection tube and the cover of the fibre cube.
3. Make sure that the thermocouple touches the specimen. Then set the duration of the experiment to 30 or 60 minutes depending on the experiment condition. However, it is necessary to exceed the setting time to 2 minutes to allow the microwave oven to reach the desired temperature.
4. Switch on the microwave oven. The temperature controller is designed in such a way that when the microwave furnace is on, then only the temperature controller can be set.
5. The temperature controller is set at 140°C or 220°C depending on the experiment condition. However, since the temperature controller fluctuates then it is important that the whole process of sintering is monitored.
6. When the temperature reaches the desired temperature, an additional stopwatch is used so that when the time of sintering reaches 30 or 60 minutes, the microwave oven can be switch off manually.

3.6.5 Measurements

3.6.5.1 Dimensional Changes

Changes in dimensions resulting from sintering represent an important field in powder metallurgy, especially with respect to large

scale production of parts with small dimensional tolerance. The fundamental process of sintering leads to a reduction in volume because of pore shrinkage and elimination. Measurements are taken before and after sintering. The diameter and height of the green compact is taken with a vernier caliper.

3.6.5.2 Density

Density is also taken before and after sintering. Figure 3.6.5.2 shows the balance analytical used to investigate the density of the specimens.

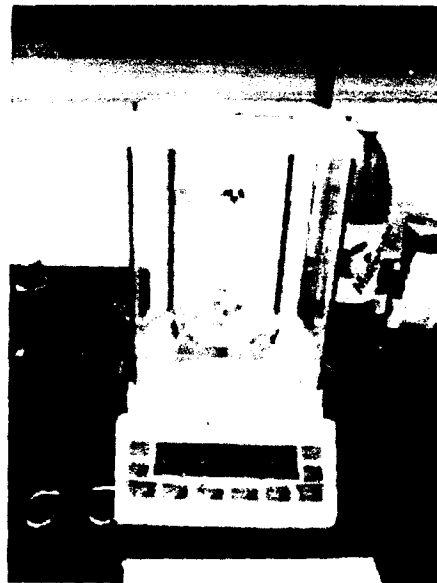


Figure 3.6.5.2 Balance Analytical

The steps below are followed to determine the density of the specimens:

1. Switch on the equipment. Briefly press scroll to get the density program
2. If "Liquid" mode is set, it will jump directly into density determination mode.
3. Select OK and place the specimen in the upper cup. As soon as the value is stable, it is stored and displayed for 2 seconds.

4. Press set when the letter g (gram) appear. This will be the weight of the specimen in air.
5. Place the same specimen in the lower cup (in the water). As soon as the value is stable, it is stored and displayed for 2 seconds.
6. Press set when the letter g (gram) appear. This will be the weight of the specimen in water.
7. Next, the density of the solid will appear. Tare balance again to gram display and ready for next determination.

3.6.5.3 Porosity

Porosity is taken after sintering. The steps below are followed to investigate the porosity of the specimens:

1. The weight of each specimen is recorded using an electronic balance before and after sintering. The weight after sintering will be the weight of dry specimen.
2. Then water is boiled on a magnetic hotplate stirrer as shown in Figure 3.6.5.3. After the water is boiled, put in the specimen and let it immersed for 30 minutes

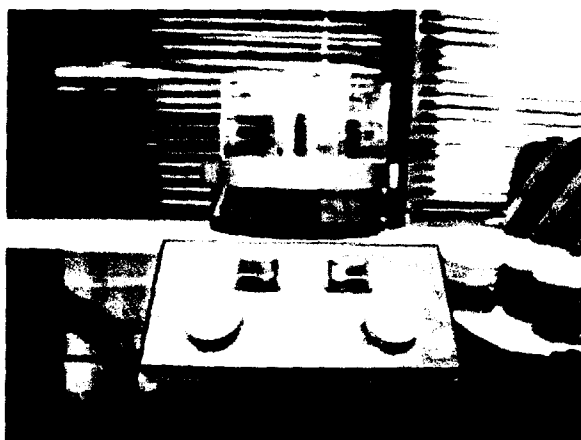


Figure 3.6.5.3 Magnetic Hotplate Stirrer

3. Let the water cool down to room temperature. Immerse the specimen in the water for at least 24 hours.
4. Then put the specimen still immersed in water in the vacuum pump desiccator as shown in Figure 3.6.5.3.1.



Figure 3.6.5.3.1 Vacuum Pump Desiccator

5. Make sure the specimen is not enclosed. Cover the vacuum pump desiccator with its lid and switch it on.
6. Adjust the knob so that the air inside the vacuum pump desiccator is suck out. Let it run for at least 30 minutes.
7. Switch off and after a few minutes, carefully open the lid. Then using the balance analytical, follow the steps of getting the density.

The weight of the saturated specimen in air and the weight of the saturated specimen in water are recorded.

3.6.5.4 Vickers Hardness Test

In the Vickers hardness test, the hardness number (HV or VHN, in kg/mm^2) is obtained on dividing the force by the surface area, calculated from the diagonal of the impression.

3.6.5.5 Microstructure Studies

The aim of this microstructure study was to get a clear image of the specimen under a microscope. The standard metallographic specimen preparation was used for microscopic study. An optical microscope linked with a computerized imaging system was used for microstructural characterization.