CERTIFICATION OF APPROVAL

SIMULATION OF SINTERING IN POWDER METALLURGY APPLICATION FOR FERROUS MATERIALS

by

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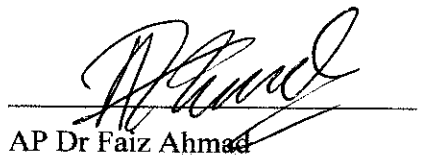

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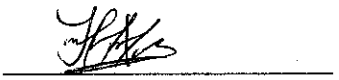
CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



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ABSTRACT

This research project is an insight of the computer simulation of sintering process used in developing parts through Powder Metallurgy. Sintering process depends on the particle size, shape, and distribution of particle size. Different types of powders will result in different packing on compaction and porosity on sintering. The objective of this research is to identify the mathematical equations that can be used for monitoring sintering process. These equations were used to generate the computer simulation to identify suitable time and temperature for sintering process.

This study was divided into two parts. First part deals with monosized particles where porosity on compaction was evaluated. Sintering parameters such as temperature and time were considered affecting on the grain growth or particle diffusion as a result of these parameters. At various temperatures, porosity was recorded and the simulation data showed that the porosity was reduced with increased in time and temperature.

In the second part, metal powders of two different sized or bimodal packing were considered. The effects of parameters such as the particle size ratio and the presence of the composition of the large particle size powder on the fractional density of the powders were investigated. The porosity of the powders was also evaluated and the data showed that the fractional porosity was reduced in less time compared to monosized particle.

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CHAPTER 1
INTRODUCTION

1.1 BACKGROUND OF STUDY

Sintering process is widely use in industrial applications nowadays especially in industries related with powder metallurgy and the ceramics manufactured. It is a main process in producing the construction materials, bricks, porcelain, abrasives and white wares. This process is not a new technology to us because it had already been discovered during the primitive age. The manufacturing process of pottery which was done by firing method made the sintering process as one of the oldest human technologies. Besides, it was found that during 3000 B.C, the Incas sintered the gold-platinum jewelry while the Egyptian already used the metal and ceramic materials [1].

Even though the sintering process had been used a long time ago, the knowledge and fundamental about this process is poorly organized. The complexities such as too many concepts, theories, characteristics of metal powders and mathematical models involved are the major factors that made the sintering studies slowly progressed. In other words, the theory has been far behind although the sintering process is widely used nowadays. The fundamental and scientifically study on this field only began after 1940s [14]. Study by Kuczynski and Frenkel at the end of 1940s was led to the first emerged of sintering quantitative models [1].

The reasons of introducing the sintering process are to give more strength and reshape the materials. Through sintering, the high strength and different shapes products can be produced from the metal powders. This process also aims to design the new microstructure of final product by controlling the grain size, allocation of particle,

sintered density and pores size. As this process is commonly being used, a technique that can predict the grain growth and porosity of particle packing is needed. This technique will help the manufacturer to control the behavior and mechanism of this process and predict the final result after undergoing the sintering process. Further, it will be helpful to produce high quality of products and reduce the cost of production. Throughout the computer simulation, the important guidelines for material design also can be obtained instead of general knowledge of microstructural growth.

Many studies on sintering and grain growth have been carried out by theoretical and experimental in order to create the simulation that really can help in assessing and optimizing sintering processes. The simulation of sintering process was first introduced in the middle of 1955 and 1965 to control and monitor the behavior and sintering mechanism [1]. It is first emerged in 1965 in which not very effective and the simulations were slow. This simulation just depicts the eventual possibilities of sintering and the results obtained are inaccurate [5]. Lot of limitations arose due to the complexity of sintering process that gives some disadvantages in creating the computer simulation.

The development of the sintering simulation always expanded whereby it then can predict the effects of time on density, temperature, pressure and size of particle for constant temperature sintering [5]. However, sintering is not an isothermal process thus the limitation of this simulation is notably not effective since sintering tend to occurs along the way to peak temperature in which for isothermal case is oppose the actual behaviour.

1.2 PROBLEM STATEMENT

The monitoring of sintering process in industry nowadays has been conventionally based on the experiences. Sometimes the trial and error method is also implemented. These methods are technically ineffective and uneconomically [6]. Thus the usage of computer simulation is more efficient and also decreases the cost of design and process description. In addition, the final product of sintering processes also can be improved.

Sintering is different from the injection molding, casting or stamping where the simulation are best to apply. The lack of volume conservation, non isothermal process and undergo the transformation of phase led the ineffective computer simulation. The forward time step also contributes to the ineffective simulation since it can result to systematic errors. Most of the computer simulations fail to estimate the error and do not reduce the error.

The sintered materials industries nowadays need the effective computer simulation of sintering to predict the sintering of product from powders. The current computer simulation on sintering faced the lack of accuracy due to some problems that related to sintering process such as changes in shape and size of particle that undergo transformation of phase.

This research project is approach to develop a simulation model for sintering process of powder compact that focused on the effects of temperature, particle size and distribution of particle.

1.3 OBJECTIVE AND SCOPE OF WORK

The main objective of this project is to simulate the mathematical equations of the sintering mechanism of metal powder particle packing.

In order to achieve the above objective, the following scope of works should be done first:

a) To study and understand the theory of sintering process.
b) To study the mathematical equations used for monosized and bimodal particle packing model.
c) To analyze the effects of sintering temperature and time for grain growth and powder compact.

1.4 SIGNIFICANT OF STUDY

This study is significant to investigate the effects of temperature and times on the sintering. This is due to different powder tends to give different result of final particles size and density. This study will simulate the sintering process for the compacts monosized particles and bimodal size particles based on mathematical equations that has been developed to predict the mechanism and behavior of sintering process.

There are a lot of changes of particle's properties upon sintering. Many characteristics of one material for instance the strength, conductivity, ductility, corrosion resistance and many more are changes significantly. These changes are the main reasons of studying the sintering process by looking at the microstructure changes in order to develop a computer simulation.

CHAPTER 2
LITERATURE REVIEW

2.1 SINTERING THEORY AND PRACTICE

The sintering process has been applied for thousands of years. One of the very first products that was produced through this process was the bricks [1]. Sintering is one of the processes in powder metallurgy that used to increase the strength of material whereby the powder materials are heated at the temperature below the melting point of the materials [4]. This process is the final step in the densification process to enhance the materials properties.

During the sintering process, the net-shaping is permitted due to the attractiveness of the particle. However, in prior to achieve the desirable finishing physical properties, the bonds between the particles must be created. When the compact powders being exposed to relatively high temperatures will result in bonding formation between the particles of the material thus the strength of this material is increased.

The formation of particle bonds is increased as the temperature is increased, longer times or the particle is small. When the temperature is increased, the greater shrinkage will occurs and the grains will growth rapidly. As the sintering time is longer, the greater expense of particles, coarsening and grain growth. Decrease in particle size led to the faster sintering process with higher impurity level. The bonds created will result in lowering the surface energy since it removed the surface area. The interparticle bonds will form along with the removal of grain boundary area [1].

The noticeable feature of sintering is the formation of necking between the contacting particles. When the particles are closed to each other, the initial stage of sintering will occurs due to weak cohesive bond at the contacts and will lead to the formation of interparticle neck vigorously. An interconnected formed during the intermediate stage whereby the smooth pore structure is recognized. This necking process will create a new grain boundary and continued until it becomes a single large particle [1].

There are two categorised of sintering process namely are solid state sintering and liquid phase sintering. When the powder compact is dense completely in a solid state at the sintering temperatures, this process is known as solid state sintering. Meanwhile, for the case of generating a liquid during the sintering process, this process is known as liquid phase sintering [14].

Since this process permits the parts to be formed, all ceramic materials and many high temperature metals are sintered to produce the desired shapes. For both of these materials, many of them are bonded by solid-state diffusion. However, the liquid phase sintering will improved the mass transport rates, densification, and microstructure coarsening due to the presence of liquid [1]. The viscosity of fluid will led to the full densification of the powder compact when the volume fraction is adequately high.

2.2 DIFFUSION PROCESS IN SINTERING

Diffusion is the most important mechanism in sintering process that leads to the changes in particle's properties. It is a mechanism of mass transport of the particle as being forced by correspondingly driving force. Two common types of mass transport mechanisms in sintering are the surface and bulk transport [10]. Surface transport includes the surface diffusion in which it gives no dimensional changes, just lead to the neck growth formation and also the evaporation-condensation process. Bulk transport comprises of volume diffusion, grain boundary diffusion, plastic flow and viscous flow whereby the densification effects can be observed.

2.2.1 SURFACE DIFFUSION

Surface diffusion involves the movement of adjacent atoms, molecules and particles at the surface of the materials. For almost of the materials, it acts as the contributor to the sintering process [1]. This kind of diffusion has three stages that lead toward the formation of neck growth. Initially, the atoms will be split from its' originally bonds. Then, these atoms will move across the surface randomly in motion before the atom reattach at an available new surface site.

The flow of mass is from surface sources to surface sinks thus the densification is not produced via the surface diffusion. Besides producing the neck growth, it also leads to the loss of surface area of the particles. The significance of the surface diffusion also decreases as the sintering process continually progress. However, this diffusion mechanism does not lead to the shrinkage of the materials [14].

2.2.2 VOLUME DIFFUSION

The movement of vacancies via a crystalline structure occurred during the volume diffusion process whereby temperature, composition and curvature are the factors that affect the rate of diffusivity [1]. This diffusion type has three ways of vacancy flow which are:-

a) *Volume diffusion adhesion* at which flow from the neck surface via the interior of the particle. No densification is occurred due to the transportation to the surface sites only.
b) *Volume diffusion densification* at which flow from the neck surface to the interparticle grain boundary. Shrinkage or densification is produced during this process.
c) *Dislocation climb* at which the vacancies being discharged by dislocations. Densification is occurred because not involves at the surface.

This diffusion process is also known as lattice or viscous diffusion.

2.2.3 GRAIN BOUNDARY DIFFUSION

Grain boundary diffusion permits the flow of mass with intermediate activation energy and form the sintered bonds by eliminate the mass at the grain boundary and relocate at the sinter bond [1]. This diffusion mechanism is important to the sintering densification of metals and compounds. Even though the presence of this mechanism is narrow, it is still and active mass transport in sintering.

2.3 EFFECT OF TEMPERATURE ON PARTICLE SIZES

There are a lot of parameters being used in order to assess or predict the result of sintering process since the entire initial properties of materials are changing. According to German (1996), the sintering time or sintering temperature is used in the measurements that will "allow examination of the sintering kinetics" [1].

Diffusion process is dependent upon the temperature. Thus, the rate of diffusion is obviously being controlled by the temperature whereby the higher temperature gives more rapid sintering. This happened because at high temperature, there will be more available sites and high number of actives atoms.

As the temperature of sintering increased, it will affect the size of particle. The higher value of temperature will result in increase of particle size with the constant sintering time. This is because as the temperature become higher, the grain growth of the particle will increase thus the size of the particle.

2.4 PARTICLE PACKING

It is very important to understand the features of particle packing since it can lead to least problems during the process. There are many types of particle packing models but the most commonly is monosized spheres. However, the bimodal packing also being considered in this project since it can give higher packing density compared to monosized model. The fractional density of the particle is the main property being concerned in the particle packing [13].

2.4.1 MONOSIZED SPHERES MODEL

The monosized spheres model is used when both of particles are in same sized. For this type of model, three types of packing that associated with monosized spheres are ordered, random loose and random dense [13]. Figures below depict the arrangement of particle for this type of model:

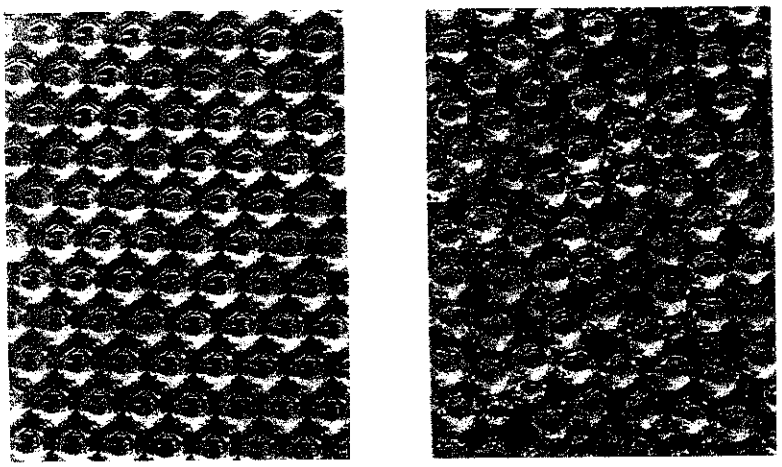

Figure 1: The Arrangement of Sphere packing of ordered (left) and random (right)

German had studied that for the solid state sintering, the final sized of grain growth will be determined by using the following mathematical equations [1]:

$$G^3 = G_0^3 + Kt \quad \text{..... (1)}$$

and $$K = K_0 \exp\left(-\frac{Q}{RT}\right) \quad \text{..... (2)}$$

where; G is the final grain size and G_0 is the initial grain size,
K is the thermally activated parameter,
K_0 is the grain boundary diffusion frequency,
t is the sintering time,
Q is the activation energy,
R is the gas constant,
T is the temperature.

Meanwhile, a model that known as LSW model had been developed by Lifshitz and Slyozov and Wagner to determine the grain growth of particles in liquid phase sintering and expressed as follows [9]:

$$G^3 = G_0^3 + K_{LSW}\, t \quad \text{..... (3)}$$

and $$K_{LSW} = \frac{64DC\Omega\gamma}{9RT} \quad \text{..... (4)}$$

where; D is the diffusivity,

C is the grain atoms solubility,

Ω is the atomic volume,

γ is the surface energy,

K_{LSW} is the parameter that developed by Lifshitz and Slyozov and Wagner.

Another equation that can be used to predict the grain growth for liquid phase sintering is

$$G^3 = G_0^3 + K_0 t \exp\left(-\frac{Q_G}{RT}\right) \quad \text{..... (5)}$$

where; Q_G is the grain growth activation energy,

K_0 is the grain boundary diffusion frequency.

This equation had been expressed by German [11].

2.4.2 BIMODAL PACKING SPHERES MODEL

The bimodal size packing model is used for a mixture of two particle sizes in which has two distinct modes in the particle size distribution. This type of packing give the higher densities that can be achieved compared to monosized particle packing type. In this packing, the large difference in particle sizes will improve the packing density since the small particles will fit in the interstitial space between the larger particles [16]. The large particle size ratio will lead to higher packing density [13]. The packing density also depends on the composition of large particle and small particle [16].

In the bimodal packing model, the weight fraction of particles and the fractional densities are considered in order to relate them with the effect of temperature towards the size of particles. Study by German [13] expressed the weight fraction as follow:

$$X_L = \frac{W_L}{W_L + W_S} \quad \text{..... (6)}$$

where; X_L is the weight fraction of large particles,
W_L is weight of large particle,
W_S is weight of small particle.

German [16] also stated that the maximum packing densities of bimodal mixtures can be calculated by using the following equations:

$$f^* = f_L + (1 - f_L) f_S \quad \text{..... (7)}$$

where; f^* is the maximum packing density,
f_L is the packing density of large particle,
f_S is the packing density of small particle.

This maximum packing density is take place at the weight fraction of large particle, X*, as expressed by follow:

$$X^* = \frac{f_L}{f^*} \quad \text{..... (8)}$$

In order to calculate the respective fractional density, f, with respect to the corresponding value of weight fraction of the large particle, X_L, the following equations being used [16]:

$$f = \frac{f_L}{X_L} \qquad \text{for } X_L > X^* \quad \text{..... (9)}$$

and $$f = \frac{f_S}{(f_s X_L + 1 - X_L)} \qquad \text{for } X_L < X^* \quad \text{..... (10)}$$

In order to relate the fractional density with the fractional porosity, the equation 11 that developed by German [1] will be used:

$$V_p = 1 - f \quad \text{..... (11)}$$

where: V_p is the fractional porosity of the particle,
f is the fractional density of the particle.

CHAPTER 3
METHODOLOGY

Prior to complete this project successfully, the methodology of the project is very important so that all the studies and works follow the project timeline smoothly.

3.1 PROCEDURE IDENTIFICATION

Below are the steps that involve throughout the completion of this study:

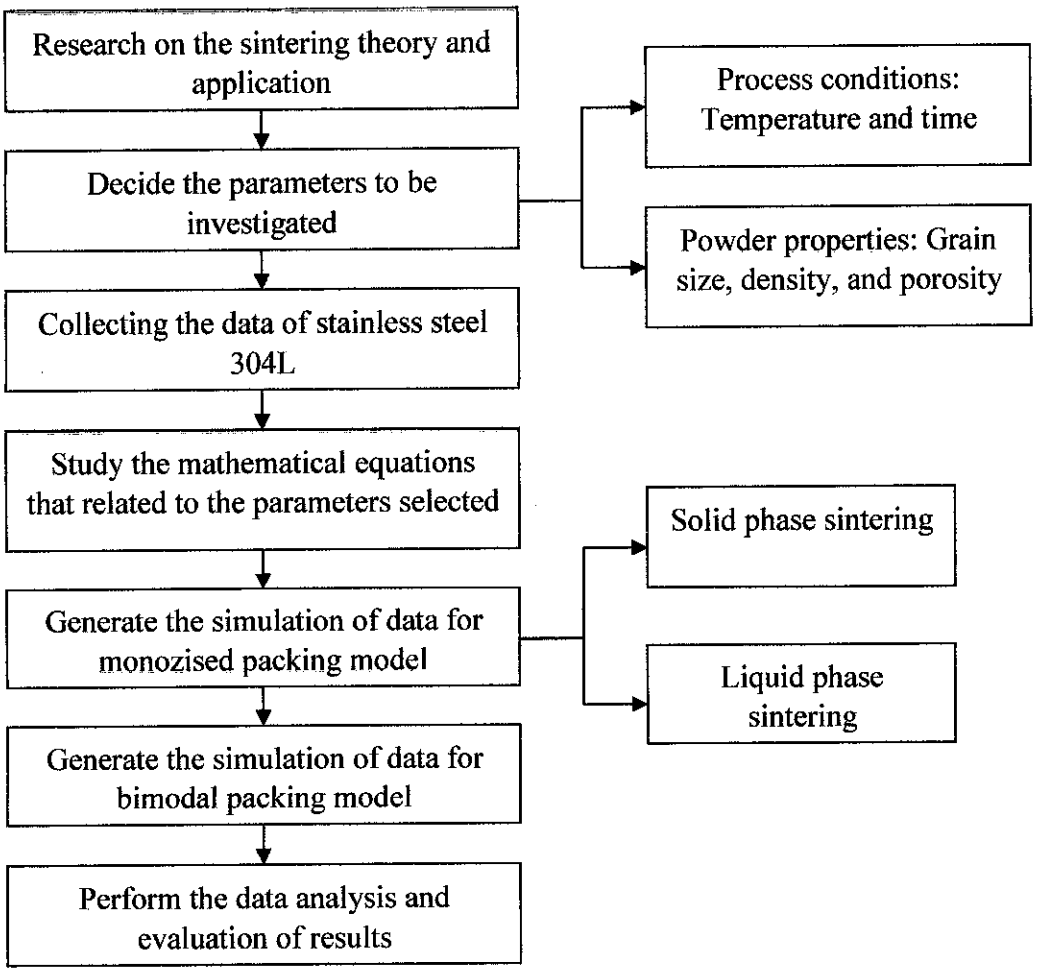

Figure 2: Process Flow Chart on the Procedure Identification

3.2 RESEARCH AND DATA COLLECTING

Research is done through some reading of the books, internet articles and journal. Throughout the research, all the information and data about sintering and computer simulation of sintering is are collected. These references also help the author to get the understanding and knowledge prior to complete this research project.

3.3 STUDY OF SINTERING PARAMATERS AND MATHEMATICAL MODEL

The study of sintering measurement techniques is important since there are too many parameters can be used. After studying all the parameters, the author will determine which technique will be used and focused in this project. After the technique of measurement is decided, the mathematical model that involved in that particular technique will be studied before the computer simulation can be generated.

The parameters that used in this research study are the diffusion involved and also the effect of temperature to the particle size. These two parameters are related to each other by the Arrhenius' Law as depicted below:

$$D = D_0 \exp\left(-\frac{Q}{RT}\right) \quad \text{..... (12)}$$

where; D is the diffusivity,

D_0 is the pre-exponential factor or diffusivity frequency,

Q is the activation energy,

R is the gas constant,

T is the temperature.

3.3.1 MATHEMATICAL MODEL FOR MONOSIZED PACKING

As discussed in the literature review part on page 10, the equation 1 and 2 will be used to determine the particle size after the solid state sintering process.

$$G^3 = G_0^3 + Kt \quad \text{..... (1)}$$

and $$K = K_0 \exp\left(-\frac{Q}{RT}\right) \quad \text{..... (2)}$$

By substituting equation 2 into equation 1, the following equation is obtained

$$G^3 = G_0^3 + \left[K_0 \exp\left(-\frac{Q}{RT}\right)\right]t \quad \text{..... (13)}$$

By using the following assumptions in Table 1 and the stainless steel's data in Table 2 on page 22, the author will use equation 12 to simulate the relationship of sintering temperature towards the final grain size.

Table 1: Assumptions Used for Simulation of Data

Properties	Value
Initial particle size (μm)	4
Range of sintering temperature, T (°C)	1000 - 1350
Sintering time (minutes)	30
Pore diameter (μm)	2
Grain boundary diffusion frequency, K_0 (m^3/s)	2×10^{-13}
Grain boundary activation energy, Q (kJ/mol)	167
The Gas Constant (J/K.mol)	8.32

For the case of liquid phase sintering, in order to relate the effect of temperature towards the particle size, the study from Lifshitz and Slyozov [9] which are the equation 3 and 4 on page 11 being studied:

$$G^3 = G_0^3 + K_{LSW}t \quad \text{..... (3)}$$

and $$K_{LSW} = \frac{64DC\Omega\gamma}{9RT} \quad \text{..... (4)}$$

So, both of the equations can be connected and the relationship between temperature and particle size can be simulated. However, in the real situation, some of these parameters cannot be applied since the usage of this equation differs compared to the real process. For example, the solubility of matrix concentration does not apply since the fraction of the solid volume is very high and grains are in contact [9].

As, the alternative way to determine the final particle size, the equation 13 being used and the relationship between the particle size or grain growth with the temperature can be obtained.

From equation $$\frac{dG}{dt} = \frac{K}{G^2} \quad \text{..... (14)}$$

where K is given by $$Kf(V_1) \propto \frac{DC\Omega\gamma}{RT} f(V_1)$$

The parameter K is also exhibit like diffusivity, D, which is depends on exponential. Thus, K is given by

$$K = K_0 \exp\left(-\frac{Q}{RT}\right) \quad \text{..... (15)}$$

where K_0 is the grain boundary diffusion frequency.

By substituting equation 13 into the equation 12, it will give

$$\frac{dG}{dt} = \frac{K_0}{G^2} \exp\left(-\frac{Q}{RT}\right) \quad \text{..... (16)}$$

In order to relate the effect of temperature towards the size of particle, the equation 16 will be integrate to form the following equation:

$$G^3 = G_0^3 + 3\int_0^t K_0 \exp\left(-\frac{Q}{RT}\right) dt \quad \text{..... (17)}$$

With the same data and assumptions that been stated in Table 1, the Equation 17 will be used in this study to generate the computer simulation of sintering looking the effect of temperature towards the particle size for liquid phase sintering.

The fractional porosity of the final size particle will be calculated using the following equation.

$$V_P = \pi\left(\frac{d_P}{G}\right)^2 \quad \text{..... (18)}$$

where; V_P is the fractional porosity
d_P is the diameter of pore,
G is the grain size of particle.

From the results obtained through the simulation, the graphs showing the relationship between the sintering temperature and sintering time toward the particle sizes will be plotted where the required temperature for the sintering process being highlighted.

3.3.2 MATHEMATICAL MODEL FOR BIMODAL PACKING

For bimodal packing model, the equation 6 on page 12 was used and being converted to the ratio of the particles in determining the final particle size:

$$X_L = \frac{W_L}{W_L + W_S} \quad \text{..... (6)}$$

where; X_L is the weight fraction of large particles,
W_L is weight of large particle,
W_S is weight of small particle.

This weight fraction then will be related to the mass fraction and finally relate it with the particle size using the following equations.

$$W = m \times g \quad \text{..... (19)}$$

and $$m = \rho \times v \quad \text{..... (20)}$$

where; W is the weight of particle,
m is the mass of particle,
g is the gravitational force,
ρ is the density of particle,
v is the volume of particle.

The equation 6 then being further modified since $W = m \times g$ and $m = \rho \times v$. So in the end this equation becomes:

$$X_L = \frac{v_L}{v_L + v_S} \quad \text{..... (21)}$$

where; X_L is the weight fraction of large particles,
v_L is volume of large particle,
v_S is volume of small particle.

But volume, $v = \frac{\pi}{6} D^3$, thus the weight fraction of the large particle is expressed by:

$$X_L = \frac{(D_L)^3}{(D_L)^3 + (D_S)^3} \quad \text{..... (22)}$$

And for X_S, weight fraction for small particles, is given by:

$$X_S = \frac{(D_S)^3}{(D_S)^3 + (D_L)^3} \quad \text{..... (23)}$$

where; D_L is the diameter of the large particle

D_S is the diameter of the small particle

By dividing the equation 22 with 23, the new equation becomes:

$$\frac{D_L}{D_S} = \sqrt[3]{\frac{X_L}{X_S}} \quad \text{..... (24)}$$

where; $\frac{D_L}{D_S}$ is the particle size ratio

As being discussed in the literature review parts on page 12 and 13, the equation 7, 8, 9 and 10 are used to study the relationship between the fractional density, the composition in terms of large particles and also the particle size ratio.

In addition, the following equation will be used to determine the relationship between particle size ratio and the fractional density that can be obtained.

$$f = \frac{0.64}{\left[1-\left(0.362-0.3615\left(\frac{D_S}{D_L}\right)^{0.7}\right)X_L + 0.995\left(\frac{D_S}{D_L}\right)^4\left(\frac{X_L^2}{1-X_L}\right)\right]} \quad \text{..... (25)}$$

Where; f is the fractional density,

X_L is the weight fraction of the large particle,

$\frac{D_S}{D_L}$ is the inverse particle ratio.

The graph that depicts the fractional density versus the particle size ratio will be plotted to show the relationship between these two parameters.

3.4 SIMULATION OF DATA

The data referred in Table 1 and 2 was use for simulation of data. This part is done after all the studies being conducted. By using the Microsoft Office Excel 2003, the simulation of all the equations and the data is generated. The graphical data then will be plotted to show the relationship between all parameters that involved in this study such as grain size, temperature, time, porosity and density.

3.5 TOOL OR DATA REQUIRED

1) Computer

   - Using the Microsoft Office Excel 2003 software to simulate the mathematical models involved.

2) Data

   - The properties of Stainless Steels 304L being used in this project as shown in the Table 2 below:

Table 2: The Properties of Stainless Steel 304L

Properties	**Value**
Composition	Fe-18Cr-8Ni
Melting Temperature (°C)	1400
Heat Capacity (J/[kg.°C])	500
Surface Energy (J/m^2)	2.2
Elastic Modulus (GPa)	193
Yield Strength (Mpa)	220
Thermal Conductivity (W/[m.°C])	16
Volume Diffusion Frequency (m^2/s)	$4x10^{-5}$
Volume Diffusion Activation Energy (kJ/mol)	280
Grain Boundary Diffusion Frequency (m^3/s)	$2x10^{-13}$
Grain Boundary Diffusion Activation Energy (kJ/mol)	167
Surface Diffusion Frequency (m^2/s)	0.5
Surface Diffusion Activation Energy (kJ/mol)	220

CHAPTER 4
RESULT AND DISCUSSION

The simulation was done based on the mathematical equations stated in the methodology part corresponding to their powder compacts packing types. The metal powder that had been used for this study is Stainless Steel 304L since this study only focus on ferrous material. After simulating the mathematical equations, all the results were tabulated and presented graphically to show the relationship of sintering temperature, time, final grain size, density, particle size ratio, and porosity.

The simulation was done separately for two parts of studies which are the monosized packing and bimodal packing model. For the monosized packing model, the relationships between the sintering temperature and time toward the final grain size been investigated. While for the bimodal packing model, the relationships between the fractional density, particle size ratio and weight fraction of the large particle were considered. The porosity of the powder compact was also investigated. The data that had been used are for the Stainless Steel 304L and involved the assumptions in order to generate this simulation.

4.1 MONOSIZED PACKING MODEL

The simulation of monosized packing model was done for two types of sintering process. The first one is for the solid state sintering. For this type of sintering, two conditions of sintering process being simulated are assumed to be as follow:

a) Sintered to specific sintering temperatures with constant sintering time
b) Sintered to specific sintering time with constant sintering temperature

The first condition used the initial particle size of 4μm stainless steel powder and was sintered up to 1350°C with 30 minutes constant time. The simulation used the Equation 13 and the results that showed the corresponding final grain size and sintered temperature was tabulated as in Table 3.

Table 3: Final Grain Size of Stainless Steel Powder Sintered for 30 Minutes.

Temperature (°C)	Final Grain Size, G (μm)	Temperature (°C)	Final Grain Size, G (μm)
1000	4.9	1180	7.7
1010	5.0	1190	8.0
1020	5.1	1200	8.2
1030	5.3	1210	8.4
1040	5.4	1220	8.6
1050	5.5	1230	8.9
1060	5.6	1240	9.1
1070	5.8	1250	9.4
1080	5.9	1260	9.6
1090	6.1	1270	9.9
1100	6.2	1280	10.1
1110	6.4	1290	10.4
1120	6.6	1300	10.7
1130	6.8	1310	10.9
1140	6.9	1320	11.2
1150	7.1	1330	11.5
1160	7.3	1340	11.8
1170	7.5	1350	12.1

Based on the above results, the graph that depicts the relationship between the sintering temperature and final grain size is plotted as shown in Figure 3. The graph in Figure 3 shows that as the temperature increased, the final grain size is also increase. This is because the formation of bonding between the grains is increased rapidly as the movement of atom is greater. The growth of these grains size eliminates the surface area and pores between the powder particles. Thus the density of the final product is increased. From the graph we can see that if the required sintering temperature which is 1300°C, the final grain size is 10.7μm. This show that the grain growth is increased rapidly and final product is denser compared to the original powder.

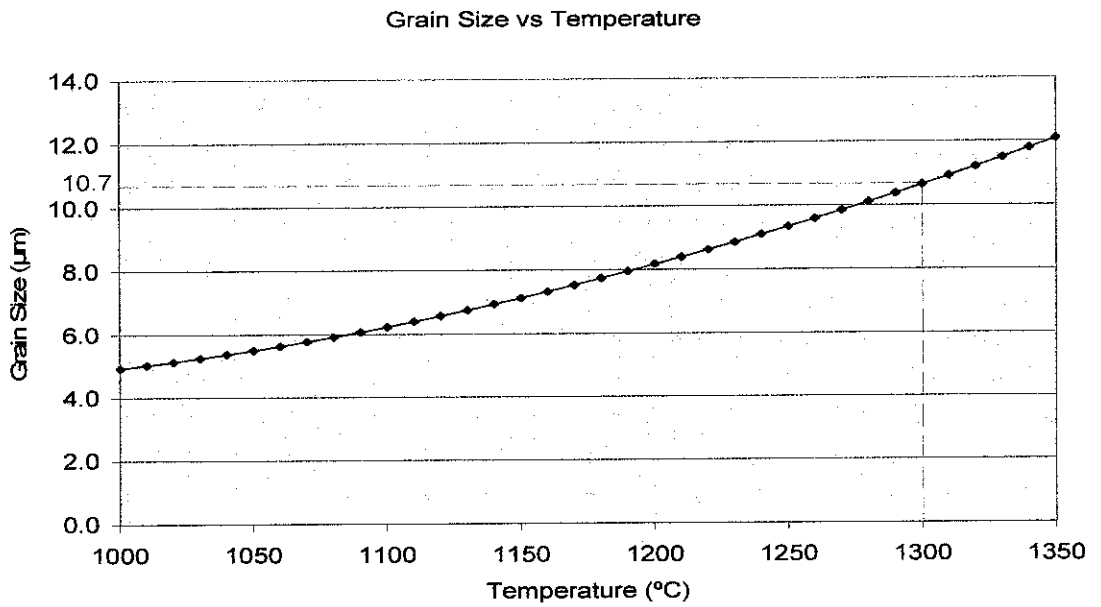

Figure 3: Grain Size versus Sintering Temperature for Solid State Sintering

For the second condition which is 4µm metal powder was sintered at two different temperatures which are at 1000°C and 1200°C for 30 minutes. The results were tabulated in Table 4 and 5.

Table 4: Final Grain Size of Stainless Steel Powder Sintered at 1000°C.

Time (min)	Final Grain Size, G (µm)	Time (min)	Final Grain Size, G (µm)
1	4.04	16	4.50
2	4.07	17	4.53
3	4.10	18	4.56
4	4.14	19	4.58
5	4.17	20	4.61
6	4.20	21	4.64
7	4.23	22	4.66
8	4.27	23	4.69
9	4.30	24	4.72
10	4.33	25	4.74
11	4.36	26	4.77
12	4.39	27	4.79
13	4.42	28	4.82
14	4.45	29	4.84
15	4.47	30	4.86

Table 5: Final Grain Size of Stainless Steel Powder Sintered at 1200°C.

Time (min)	Final Grain Size, G (μm)	Time (min)	Final Grain Size, G (μm)
1	4.28	16	6.66
2	4.53	17	6.77
3	4.75	18	6.87
4	4.96	19	6.98
5	5.15	20	7.07
6	5.32	21	7.17
7	5.49	22	7.26
8	5.65	23	7.35
9	5.79	24	7.44
10	5.93	25	7.53
11	6.07	26	7.61
12	6.20	27	7.69
13	6.32	28	7.77
14	6.44	29	7.85
15	6.55	30	7.93

Based on Table 4 and 5, the graphs of grain size versus the sintering time were plotted to depict the relationship between these two parameters.

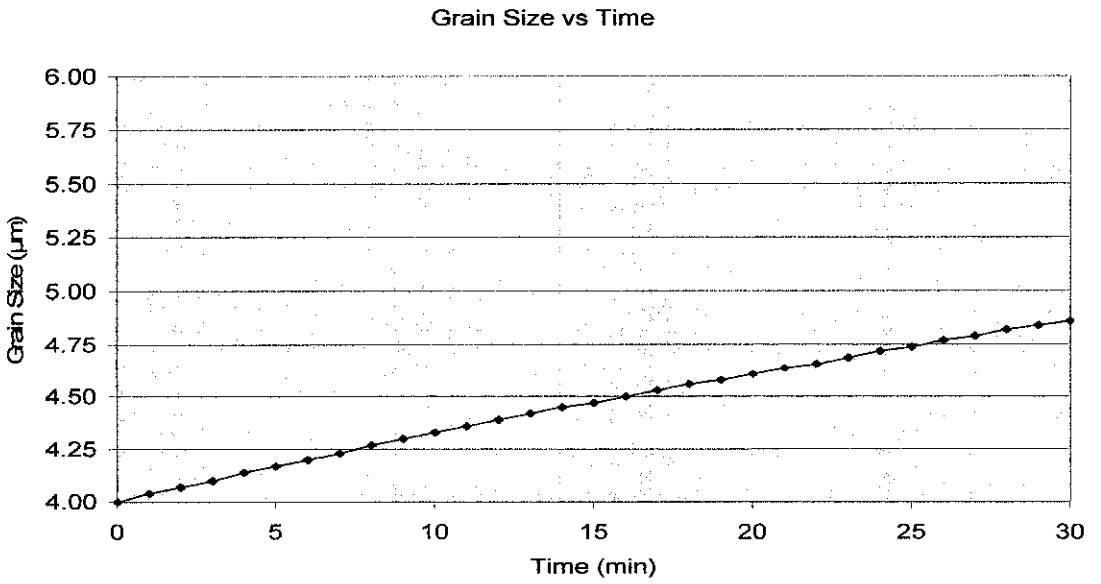

Figure 4: Grain Size versus Sintering Time at 1000°C.

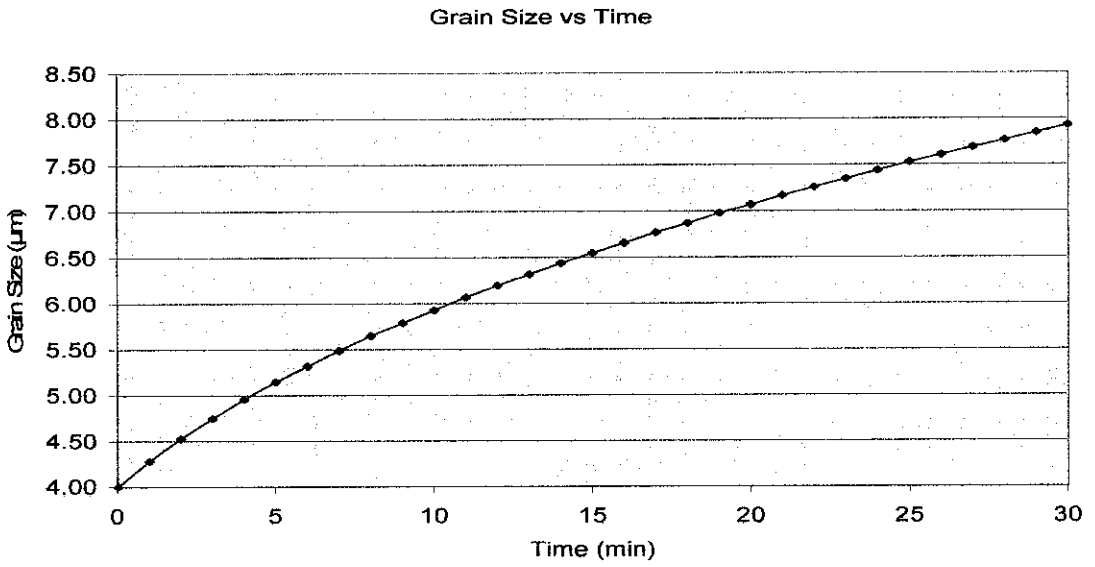

Figure 5: Grain Size versus Sintering Time at 1200°C.

In Figure 4 and 5, as the sintering time increased, the final grain size also increased. This is because as the time is increased, the grain is allowed to grow more thus the greater expense of the particle occurred.

If the comparison of both of these figures is made, the final grain size is rapidly increased for the higher sintering temperature than the lower sintering temperatures even though the sintering time is same. This phenomenon happened due to the formation of bonds is faster for the high sintering temperature.

For the liquid phase sintering the simulation is done by using the Equation 17. The initial grain size of 4µm stainless steel powder was sintered up to 1350°C with 30 minutes constant sintering time. The following are the results of simulation as tabulated in Table 6.

Table 6: Final Grain Size of Stainless Steel Powder for Liquid Phase Sintering

Temperature (°C)	Final Grain Size, G (μm)	Temperature (°C)	Final Grain Size, G (μm)
1000	6.166	1180	10.817
1010	6.355	1190	11.145
1020	6.553	1200	11.481
1030	6.760	1210	11.824
1040	6.976	1220	12.173
1050	7.200	1230	12.528
1060	7.432	1240	12.891
1070	7.672	1250	13.259
1080	7.920	1260	13.634
1090	8.176	1270	14.016
1100	8.440	1280	14.404
1110	8.712	1290	14.798
1120	8.991	1300	15.198
1130	9.227	1310	15.605
1140	9.571	1320	16.018
1150	9.871	1330	16.436
1160	10.179	1340	16.861
1170	10.495	1350	17.292

The grain size versus the sintering temperature graph is plotted using the above results to show the relationship between those two parameters.

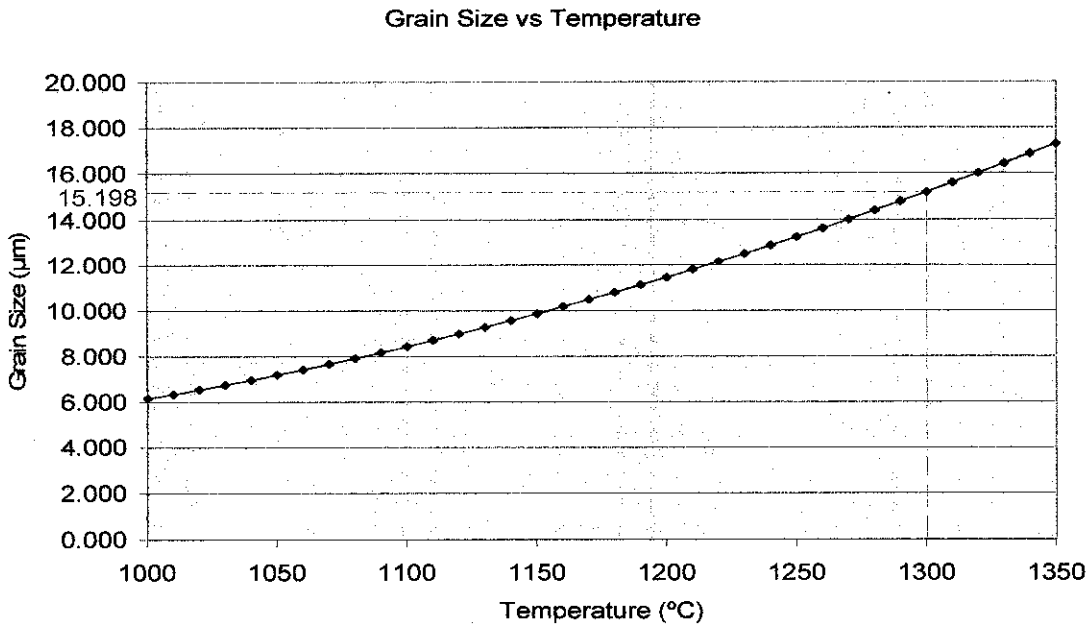

Figure 6: Grain Size versus Sintering Temperature for Liquid Phase Sintering

From the Figure 6, the final grain size of stainless steel powders increased as the sintering temperature increase. At the sintering temperature is 1300°C, the corresponding final grain size of particles is 15.198μm. It can be seen that the final grain size produced by liquid phase sintering is larger compared to the solid state sintering. This is because the presence of liquid in this process increased the flow of mass transfer to form the sintered bonds and the grain growth of the metal powders.

The Equation 18 will be used to show the relationship between the grain size and the fractional porosity. For this simulation, the first condition for solid state sintering which is the 4μm of stainless steel powder is used. This powder is sintered up to 1350°C for 30 minutes. The size of pore diameter is assumed to be 2μm. Following are the tabulated result of this simulation.

Table 7: The Fractional Porosity of Stainless Steel Powder Sintered at the Corresponding Grain Size.

Grain Size, G (μm)	Fractional Porosity, Vp	Grain Size, G (μm)	Fractional Porosity, Vp
4	0.7855	8.5	0.1740
4.5	0.6206	9	0.1552
5	0.5027	9.5	0.1393
5.5	0.4155	10	0.1257
6	0.3491	10.5	0.1140
6.5	0.2975	11	0.1039
7	0.2565	11.5	0.0950
7.5	0.2234	12	0.0873
8	0.1964		

The graph that depicts the relationship between the grain size and the fractional porosity of the powder is plotted as shown in Figure 7.

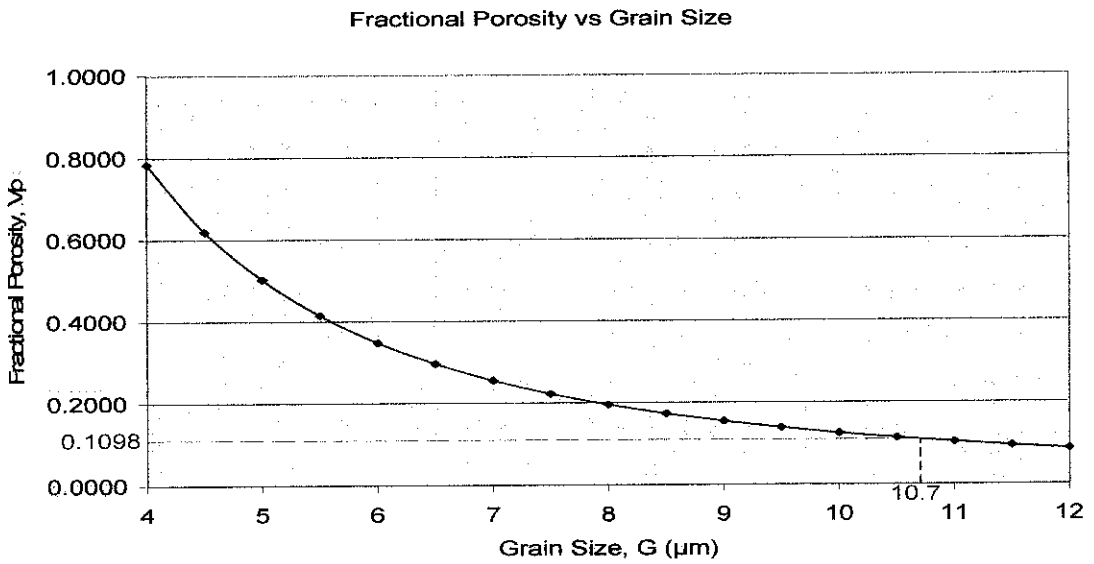

Figure 7: Fractional Porosity versus Grain Size for Solid State Sintering

In the Figure 7 above, the fractional porosity of the metal powder is decreased as the grain size is increased. From this figure, the author can conclude that the fractional density is inversely proportional with the grain size. At the grain size equal to 10.7μm, the corresponding fractional porosity is 0.1098. So, the reduced in the percentage of porosity which is about 67% showed that the final density of the powder is increased.

4.2 BIMODAL PACKING MODEL

The simulation of bimodal packing model is looking to the effects and relationship between the particle size ratio, fractional packing density and the weight fraction of the large particle. By using stainless steels as metal powder, the Equation 25 is used to generate the simulation for the relationship between the particle size ratio and the fractional density. The size of smaller particles is 5μm while for the large particle the size is in range 10μm till 500μm. The weight fraction or composition of the large particle that been used is 70%. Table 8 below tabulated the results of the simulation.

Table 8: The Optimum Fractional Density with the Corresponding Particle Size Ratio

Small Particle Size (μm)	Large Particle Size (μm)	Particle Ratio, DL/Ds	Fractional Density, f
5	5	1	0.6400
5	10	2	0.6532
5	25	5	0.7799
5	50	10	0.8094
5	100	20	0.8272
5	150	30	0.8344
5	200	40	0.8385
5	250	50	0.8412
5	300	60	0.8430
5	350	70	0.8445
5	400	80	0.8456
5	450	90	0.8465
5	500	100	0.8473

The graph of fractional density versus particle size ratio is plotted as depicted in Figure 8. As the particle size ratio is increased, the fractional density of the particle also increased. However, when the ratio is larger than 60, it is obviously that the fractional density is achieved its saturated point and no longer increased. The larger changes of fractional density can be seen in the range of 5 up to 10.

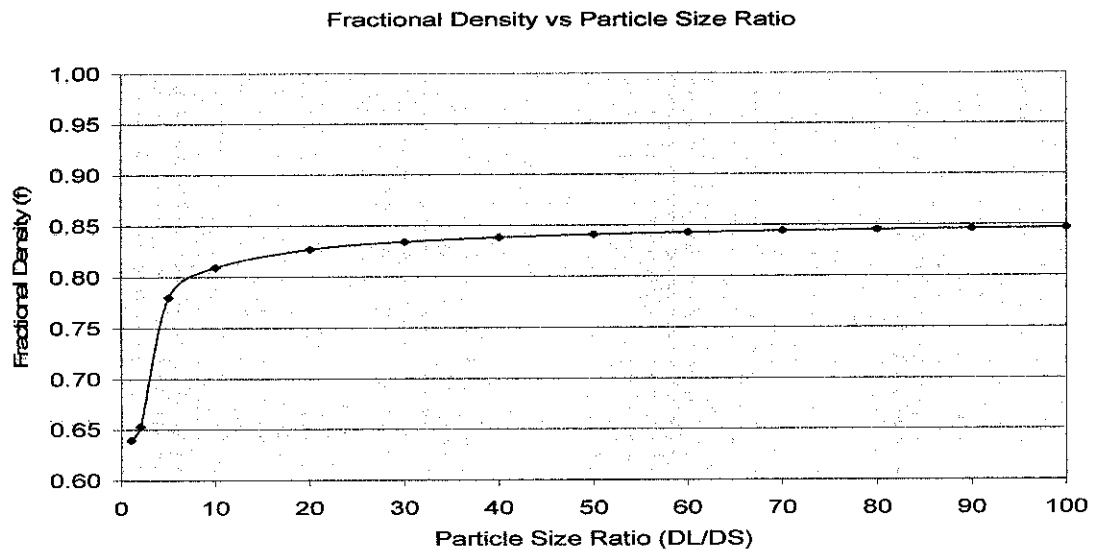

Figure 8: Fractional Density versus Particle Size Ratio for Bimodal Packing

By using the Equation 7, 8, 9 and 10, the simulation that analyzes the relationship between the composition or weight fraction of the large particle, X_L, towards the packing density is generated. The simulation assumed that the maximum packing density is achieved at the 70% of weight fraction of large particle, X^*. In order to determine the fractional density of the particle at the X_L more than X^*, the Equation 9 is used while for X_L less than X^*, the Equation 10 is used. The values of packing density at the corresponding weight fraction of large particle are tabulated as in the Table 9.

Table 9: The Fractional Density of Powders at the Corresponding Weight Fraction of the Large Particle.

Weight Fraction of large particle, X_L	Fractional Density, f
0	0.5602
0.1	0.5860
0.2	0.6143
0.3	0.6454
0.4	0.6798
0.5	0.7181
0.6	0.7610
0.7	0.8094
0.8	0.7082
0.9	0.6295
1	0.5666

The graphical data of the above results is plotted as illustrated by Figure 9 below. The maximum packing density is achieved when the majority of the large particles are used than the small particles. This is because, the terminal region between the larger particles will be occupied by the smaller particles, thus improved the density of the particles.

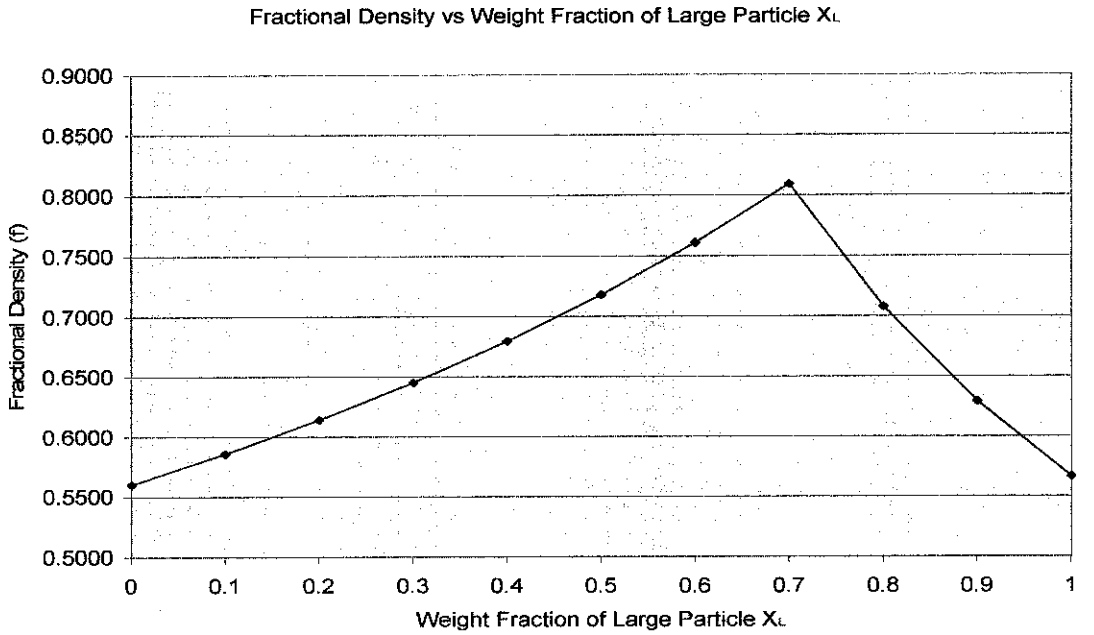

Figure 9: Fractional Density versus the Composition of Large Particles for Bimodal Packing

The final part of this simulation is to relate the packing density with the porosity. The Equation 11 is used to show the relationships of both parameters. The results of the simulation are tabulated in the Table 10 as follow.

Table 10: The Fractional Porosity of Powders at the Corresponding Weight Fraction of the Large Particle.

Weight Fraction of large particle, X_L	Fractional Density, f	Fractional Porosity, V_p
0	0.5602	0.4398
0.1	0.5860	0.4140
0.2	0.6143	0.3857
0.3	0.6454	0.3546
0.4	0.6798	0.3202
0.5	0.7181	0.2819
0.6	0.7610	0.2390
0.7	0.8094	0.1906
0.8	0.7082	0.2918
0.9	0.6295	0.3705
1.0	0.5666	0.4334

The graph that depicts the relationship between the fractional density with the fractional porosity at the corresponding weight fraction of the large particle is illustrated by Figure 10 below.

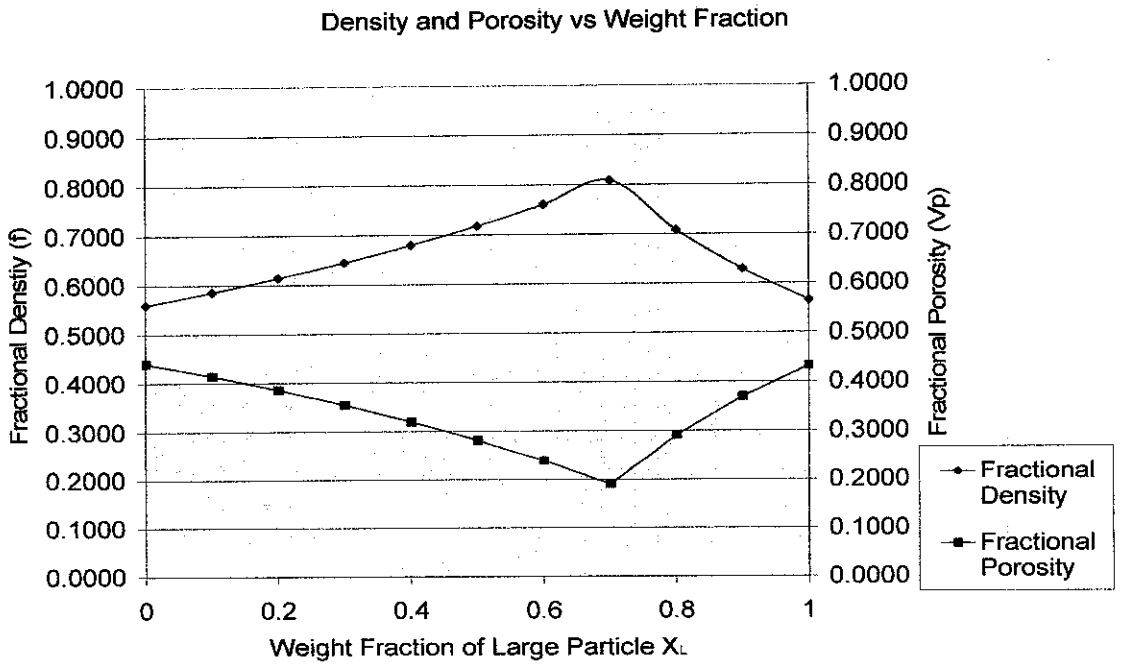

Figure 10: Fractional Density and Porosity versus the Composition of Large Particles for Bimodal Packing

The results show that the porosity is higher when the fractional density is lower. Thus, the greater density will result less terminal region between the particles. Thus, the denser particle can be produced at the higher packing density of the metal powders.

Besides, the graph also shows that the percentage of porosity is reduced for the bimodal packing model while for the monosized model the percentage of porosity is high which is occurred when weight fraction is 0 and 1. The bimodal packing model has the low porosity because the spaces between the large particles were fitted by the small particles. This will make the density of the powders increased thus reduce the porosity of the powders.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Many of the current computer simulations on the sintering process nowadays are lack of accuracies due to the presence of the complexities. Even though much effort had been exerted in this field, the available information on sintering process and the application of computer simulation is still inadequate. The manufacturers still used past companies' experienced and tend to implement the try and error method to control the microstructural growth and sintering mechanism.

The study has been completed parallel with the objective and the timeline that had been constructed. This study is done to generate the simulation of data on the sintering process by using the mathematical equations that related to the parameters that being investigated. From the studied done, the proposed project can help in improving the sintering computer simulation since the simulation only deals with parameters needed.

The study is focused on determining the final grain size and final packing density of the metal powders with respect to the sintering temperature, sintering time, the particle size ratio and the weight fraction of the large particle. The study was looking into two types of packing model which are the monosized and bimodal packing model. The effects of emperature and time toward the final particle size and porosity of powders were considered for the monosized model. For bimodal, the effects of particle size ratio and weight fraction of large particle on the density and porosity of powders were investigated.

For the monosized packing model, the final grain size increased rapidly as the metal powder has been sintered to the higher temperature and longer sintering time for both solid state and liquid phase sintering. It is also shown that the greater grain size can be obtained in liquid phase sintering compared to solid state sintering due to presence of liquid. For the solid state sintering, it is found that the percentage of porosity was reduced about 67% when sintered to 1300°C whereby the grain size of the particles increased from 4μm to 10.7μm.

The lower porosity can be obtained at the highest fractional density for the case of bimodal packing. This led to the denser final product. The study also shown that as the particle size ratio become larger, the packing density will increase until it saturated point approximately when the particle size ratio larger than 60.

5.2 RECOMMENDATIONS

For the future work of this simulation of data, the experimental work on sintering should be done since this study used lot of assumptions. By performing the experiment, the accuracy of the data simulation is better than theory itself. Besides, many conditions of the process can be set up and investigate prior to generate the best data simulation. The comparison of result between the theory and the experimental work can be done and the errors can be estimated.

Since this study only covers the stainless steel powders, further study on the different metal powders also can be done so that the comparison of the results can be made and analyzed. It is also useful in determining whether the simulation is suitable for any metal powders for powder metallurgy applications.

The differential equation techniques such as Runge Kutta and Laplace Transform also can be applied in generating more accurate equations thus lead to more accurate simulation. The simulation can be generated by using the MATLAB software since it can deals with the iteration problems.

CHAPTER 6
REFERENCES

1. German, R.M, 1996, *Sintering Theory and Practice,* New York, John Wiley & Sons.
2. Stojanovic, B.D, Skorokhod, V.V and Nikolic, M.V, 1999, *Advanced Science and Technology of Sintering,* New York, Kluwer Academic.
3. Rahaman, M.N, *Ceramic Processing and Sintering,* New York, Marcel Dakker.
4. Braginsky. M, Tikare, V and Olevsky, E. 6 Aug 2004 from http://www.engineering.sdsu.edu/~olevsky/ijss02-05.pdf
5. German, R.M. 2002 from www.cavs.msstate.edu/publications/2002-24.pdf
6. Prado, J.M, Bejarano, A and Riera, M.D. 2003 from www.elsevier.com/loacte/jmatprotec
7. German, R.M, *Computer Modeling of Sintering from* http://www.cisp.psu.edu/pdf/tech_briefs/ComputerModelSintering.pdf
8. Rawlings, A.J, Kopech H.M, Rutz, G.H. 1997 from http://www.hoeganaes.com/Publications/55.pdf
9. Park, S.J, Martin, J.M, Guo, J.F, Johnson, J.L and German, R.M, 2006, *Grain Growth Behavior of Tungsten Heavy Alloys Based on Master Sintering Curve Concept.*
10. Postula, A, Abramson, D and Logothetis, P, The Design of a Specialised Processor for the Simulation of Sintering from http://www.csse.monash.edu.au/~davida/papers/sinter.pdf

11. German, R.M, 2006, *Establishment of the Scientific Underpinnings in Powder Injection Molding and Liquid Phase Sintering.*

12. Olevsky, E.A, Shoales, G.A and German, R.M, 2000, Temperature Effect on Strength Evolution under Sintering.

13. German, R.M, 1989, *Particle Packing Characteristics,* New Jersey, Metal Powder Industries Federation.

14. Joong, S and Kang, L, 2005, *Sintering: Densification, Grain Growth & Microstructure*, Oxford, EL SEVIER.

15. German, R.M, Messing, G.L and Cornwall, R.G, 1996, *Sintering Technology,* NewYork, Marcel Dekker Inc.

16. German, R.M, 1992, *Metallurgy Transactions A: Prediction of Sintered Density for Bimodal Powder Mixtures.*

APPENDIX

APPENDIX 1

Gant Chart

- First Semester

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic															
2	Perform Research and Study of the Project															
3	Work on preliminary report															
4	Submission of Preliminary Report															
5	Project Work															
6	Work on Progress Report															
7	Submission of Progress Report															
8	Project work continues															
9	Working on Interim Report															
9	Submission of Interim Report Final Draft															
10	Oral Presentation															

- Second Semester

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	SW	EW
1	Project Work Continue																
	-Study on Monosized Model																
2	Submission of Progress Report 1																
3	Project Work Continue																
	-Working on Progress Report 2																
4	Submission of Progress Report 2																
5	Project work continue																
	-Study on Bimodal Model																
6	Submission of Dissertation Final Draft																
7	Oral Presentation																
8	Submission of Project Dissertation (Hardbound)																