CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction to Taguchi parametric robust design

Previous experimental approaches, since the invention of experimental design in the 1930s, did not consider noise factors as causes of quality variation. It is very difficult or very expensive to control or eliminate causes of variation which come from noise factors. Therefore, a different approach is required if variation of result quality is to be reduced. This approach was termed parameter design by Taguchi and this design is used to improve quality without controlling or eliminating causes of variation, and to make the results robust against noise factors (Sung, 1996).

In general, parameter design has the following characteristics. First, parameter design classifies factors which affect quality characteristics into two groups: control factors and noise (or uncontrollable) factors. A factor-characteristic relation diagram is shown in Figure 2.1. Note that 'system' here means a given experimental situation.

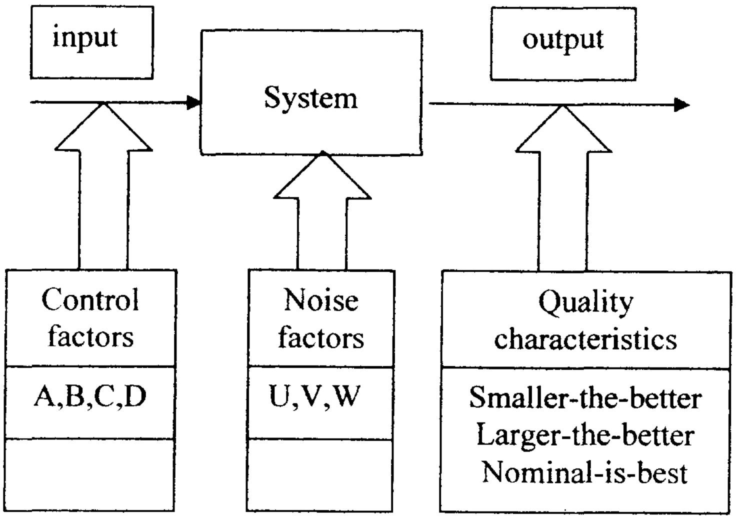

Figure 2.1 Factor-characteristic relation diagram

Second, parameter design generally uses two OAs: for the control factors, an OA which is called an 'inner array' or 'matrix of design variables'; and for the noise factors, another OA which is called an 'outer array' or 'matrix of uncontrollable factors'. Table 2.1 shows an experimental layout with an inner array (L_8) for control factors, and an outer array (L_4) for noise factors.

Type of array	Inner array (L_8)							Outer array (L_4)				
	Control factor assignment and column number							Raw data				SN ratio
								Experiment no.				Noise factor assignment
								1	2	3	4	
Experiment number	A	B	C	D	F	e	e	0	0	1	1	U
	1	2	3	4	5	6	7	0	1	0	1	V
								0	1	1	0	W
1	0	0	0	0	0	0	0	y_{11}	y_{12}	y_{13}	y_{14}	SN_1
2	0	0	0	1	1	1	1	y_{21}	y_{22}	y_{23}	y_{24}	SN_2
3	0	1	1	0	0	1	1	y_{31}	y_{32}	y_{33}	y_{34}	SN_3
4	0	1	1	1	1	0	0					
5	1	0	1	0	1	0	1					
6	1	0	1	1	0	1	0					
7	1	1	0	0	1	1	0					
8	1	1	0	1	0	0	1	y_{81}	y_{82}	y_{83}	y_{84}	SN_8

Table 2.1 Basic structure of parameter design

2.2 Previous works that used the Taguchi method

There are a lot of works that used the Taguchi parametric robust design approach in their researches. However, only a few of them is of interest in this particular study because of its relevance. Ji, Loh, Khor and Tor (2000) report the use of Taguchi method in characterizing and optimizing the process factors for sintering water-atomized 316L stainless steel. The objective of the experiments was to determine the effects of sintering factors; sintering temperature, heating rate, sintering time and sintering atmosphere on the final density and the optimum set of factors that would maximize the final density. The various factors were assigned to an L_9 orthogonal array. For the four sintering factors, each factor is designed with three levels and each experiment was conducted with four replications.

From the analysis of the experimental results using the Taguchi method, they found out that the sintering atmosphere has the most significant effect on the sintered density. The highest yield of the average density could be attained at the combined settings of sintering temperature of 1250°C, heating rate of 20°C min^{-1}, sintering time of 90 min and sintering in vacuum.

Another work that used the Taguchi method is Chua, Lu, Lai and Wong (2005) that investigated the effects of additives on the properties of doped lead zirconia titanate (PZT) processed by low-temperature sintering. Three factors with three levels of each factor are chosen and Taguchi's L_9 orthogonal array was used. The main additives such as MnO_2, $Bi_4Ti_3O_{12}$ and barium titanium silicate ($Ba_2TiSi_2O_8$) with different percentages are added to alter the properties of doped PZT. The results revealed that the properties of doped PZT ceramics sintered at 1050°C were strongly influenced by the additives with MnO_2 has the most significant effect.

A work by Kim, Park and Kim (2004), proposed a method for finding out the optimum level of each factor under study. According to this method, the optimum level for each factor will be at which the average of the "iso-level" value of S/N ratio is maximum. The average iso-level value is determined from the values of S/N ratios at low and high levels of the factors. This method of finding out the optimum conditions will be used in this study.

These three works although, not directly related to the microwave sintering of tin base alloys, shows how the Taguchi parametric robust design method influences the findings of their work.

2.3 Introduction to powder metallurgy

Powder metallurgy is a process in which fine metal powders are compacted into intricate shapes. The first known product from this process is the tungsten filament of incandescent light bulbs made early in the 20th century. More recently the powder metallurgy process has been used to produce gears, cams, bushings, cutting tools, piston rings, and valve guides for automotive engines, and aircraft parts such as jet engine disks. The process consists of several steps, including powder production, blending, compaction, sintering and finishing operations.

2.3.1 Blending or mixing

Blending the metal powders is necessary to provide a uniform distribution of powder size, for mixing powders of two metals to make an alloy product, or for mixing lubricants that improve flow of powder metal into dies. Blending is usually done in mechanical mixers under a controlled environment, such as inert gas, or in liquid lubricants. Various variables in the powder mixing process have been highlighted by Hausner. They are:

a) Type of mixer
b) Volume of the mixer
c) Geometry of the mixer
d) Inner surface area of the mixer
e) Constructional material and surface finish of the mixer
f) Volume of the powder in the mixer before mixing

g) Volume of the powder in the mixer after mixing
h) Volume ratio of component powders
i) Volume ratio of mixer to powder
j) Characteristics of component powder
k) Type, location and number of loading and emptying devices
l) Rotational speed of mixer
m) Mixing time
n) Mixing temperature
o) Mixing medium (gaseous or liquid)
p) Humidity when mixing in air

Mixing efficiency is best when the powder volume is about 50% to 60% of the mixer volume. Optimum mixing time may be from between 5 to 30 minutes but this can be determined only by experience with a given mixture in a particular mixer (Upadhyaya, 1997).

2.3.2 Compaction

During the compaction step, powders are pressed into shape using hydraulic or mechanically activated presses. The mass density increases and good particle-to-particle contact is achieved in this step. As the compacting pressure increases, the density approaches the theoretical value. After going through the compacting stage, the workpiece is known as a green compact and has the strength necessary for the next processing step.

The compaction of metal powders has the following major functions:

a) to consolidate the powder into desired shape

b) to impart, to as high degree as possible, the desired final dimensions with due consideration to any dimensional changes resulting from sintering

c) to impart the desired level and type of porosity

d) to impart adequate strength for subsequent handling

Several approaches exist for achieving these goals. In general, the techniques can be categorized as (Upadhyaya, 1997)

a) continuous vs discontinuous process
b) pressures- high vs low
c) compaction velocity - high vs low
d) temperature - room to elevated temperature
e) uniaxial vs hydrostatic pressures

2.3.3 Sintering

Sintering is an important step in powder densification. Sintering may be considered the process by which an assembly of particles, compacted under pressure or simply confined in a container, chemically bond themselves into a coherent body under the influence of an elevated temperature. The driving force for sintering resides in the enormous surface energy stored in powders due to their high surface-area-to-volume ratio. In fact, the stored energy may be a danger during storage because of its potential to explode. During sintering, the green compacts are heated to temperatures between 70 and 90% of their absolute melting temperature of the major constituent to get high diffusion rates along the powder boundaries (Schaffer, 1999).

Much of the difficulty in defining and analyzing sintering is based on the many changes within the material that may take place simultaneously or consecutively. Densification or shrinkage of the sintered part is very often associated with all types of sintering. However, sintering can take place without any shrinkage; expansion or no net dimensional change is quite possible. Sintering is a complex process and for any given metal and set of sintering conditions there are likely to be different stages, driving forces and material transport mechanisms associated with the process.

Various stages of sintering can be grouped in the following sequence (Upadhyaya, 1997):

a) initial bonding among particles
b) neck growth
c) pore channel closure
d) pore rounding
e) densification or pore shrinkage
f) pore coarsening

2.4 Introduction to Microwave Heating

To the average consumer, the term "microwave" connotes a microwave oven, which is used in many households for heating food. As shown in Figure 2.4, a microwave oven is a relatively simple system consisting of a high-power source, a waveguide feed, and the oven cavity.

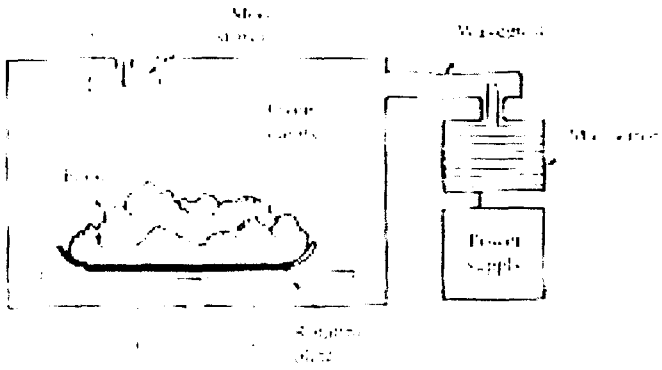

Figure 2.4 A microwave oven (Picture from Scott, Allan W. (1993) Understanding Microwaves)

The source is generally a magnetron tube operating at 2.45 GHz; its power output is usually between 500 and 1500 W (Scott, 1993). The oven cavity has metallic walls, and is electrically large. To reduce the effect of uneven heating caused by standing waves in the oven, a "mode stirrer," which is just a metallic fan blade, is used to perturb the field distribution inside the oven (Scott, 1993).

2.4.1 Microwave heating mechanism

Microwaves are used for industrial heating and for heating and cooking food in a microwave oven. In a conventional furnace a gas or charcoal fire, or an electric heating element, generates heat outside of the material to be heated. The outside of the material then gets heated by convection and the inside of the material by conduction. In microwave heating, by contrast, the inside of the material gets heated first. The process through which this occurs involves the resonance of water molecules and conduction losses in materials with large loss tangents(Scott, 1993). An interesting fact is that the loss tangents of many material decrease with increasing temperature, so that microwave heating is to some extent self-regulating. The result is that microwave heating generally gives faster and more uniform heating of material, as compared with conventional heating. The efficiency of a microwave furnace, when defined as the ratio of power converted to heat (in the material) to the power supplied to the oven, is generally less than 50%; this is usually greater than the heating efficiency of a conventional furnace, however (Scott, 1993).

Another way of explaining microwave heating mechanism is to see it in terms of wavelengths. Most heating and cooking is done with infrared radiation, which is commonly called "heat" and occurs at short wavelengths in the frequency range between microwaves and light. The short wavelengths of infrared radiation cannot penetrate materials such as food, so the infrared radiation is absorbed on the surface and conducted into the inner part of the material. Normally the material to be heated is a poor thermal conductor, so the heating is a slow process.

In contrast, the longer wavelength of microwaves can penetrate the entire material and heat it from the inside out. In a microwave oven the microwaves are absorbed by water in the food, and the food is uniformly heated throughout its cross section.

The frequency for microwave cooking and heating is 2450 MHz (Pozar, 1990). The wavelength is long enough to penetrate the material, yet short enough so that the microwaves can fit into a moderate size enclosure such as a home microwave oven.

Microwaves are a form of electromagnetic radiation and they heat substances that are polar and/or ionic in nature (http://www.organische-chemie.ch/OC/Fokus/Juni3-2003.htm). Like all electromagnetic radiation, microwave radiation can be divided into two components, one electric field and one magnetic field component. The electric field component is responsible for the heating. This component affects heating via two major mechanisms – dipolar polarization and ionic conduction.

A third mechanism - interfacial polarization - occurs, although this is often of limited importance.

2.4.1.1 Dipolar polarization mechanism (http://www.organische-chemie.ch/OC/Fokus/Juni3-2003.htm)

A dipole is sensitive to electric fields and will attempt to align itself with the field as shown in Figure 2.4.1.1. The applied field provides the energy for this rotation. "The right frequency" of the applied irradiation is when the dipoles have time to respond to the alternating electric field and therefore rotate, but the rotation does not precisely follow the field. This gives rise to a phase difference. This phase difference causes energy to be lost from the dipole, by molecular friction and random collisions, giving rise to dielectric heating.

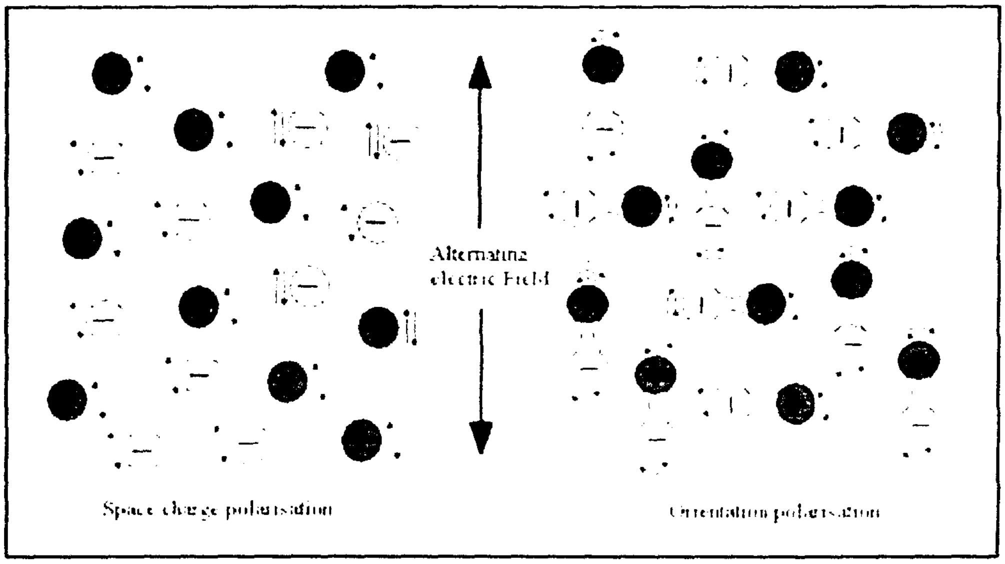

Figure 2.4.1.1 . Molecular oscillations of polarizable substances under the influence of an alternating electric field (Picture taken from http://www.staffs.ac.uk/schools/engineering_and_technology/downlevel/research/research_students/peter_wardle.htm)

2.4.1.2 Ionic conduction mechanism (http://www.organische-chemie.ch/OC/Fokus/Juni3-2003.htm)

This interaction is much stronger in regards to the heat generating capacity. Ions in the sample will move in the solution under the influence of an electric field, resulting in expenditure of energy and increased collision rate, converting the kinetic energy to heat.

2.4.1.3 Interfacial Polarization

(http://www.ipm.virginia.edu/process/PVD/Pubs/thesis4/chapter2.pdf)

In interfacial or space-charge polarization, mobile charge carriers in a heterogeneous material are accelerated by an applied field until they are impeded by and pile up at physical barriers. This build up of charge dictates the polarization of the material. Grain/phase boundaries and free surfaces are common barriers.

The polarization resulting from the mechanisms above is strongly influenced by frequency. The individual mechanisms have varied dependences of their polarization upon frequencies. In general the larger the masses involved, the slower the response upon application or removal of a field and consequently the relaxation frequency is lower. For example, ionic polarization mechanisms undergo resonance in the optical and far infrared wavelengths respectively.

2.5 Previous works on microwave sintering

Microwave sintering is a method of heating that involves energy conversion which is different from the conventional sintering that concerns energy transfer. In microwave sintering, the heat is generated internally within the material instead of originating from external sources. Microwave sintering is much more uniform at a rapid rate and hence results in reduction of processing time and energy consumption.

The recent development in the fields of microwave sintering have progressed due to its potential use for a wide range of materials from wood, bacon and potato chips to rubber, ceramics and semiconductors as reported by Clark and Sutton (1996), Schiffman (1995), Katz (1992) and Sutton (1992, 1989). However, it was the Microwave Research Group at the Materials Research Institute of the Pennsylvania State University that first demonstrated very rapid sintering in time intervals varying from 3-20 min on many traditional and advanced ceramic materials such as alumina, mullite and hydroxyapatite. This is reported by Roy, Agrawal, Cheng and Mathis (1997) and reveals transparency and almost full density.

Review of the research work reveals that much work has been done on oxide ceramics and semi-metals like carbides and nitrides. The applicability of microwave sintering to metals has been overlooked because most metals are known to reflect microwaves (http://www.azom.com). Roy *et al.* (2000) discussed the use of microwave sintering and noted that few experiments have been done with metal powders. Green laboratory and commercial compacts was microwave sintered, typically, at 1100 to 1300°C for 5 to 30 minutes. The sintered compacts were reported to have uniformly distributed porosity with properties improved in comparison with conventionally process materials.

Rodiger, Dreyer, Gerdes, and Willert-Porada (1998) reported that sintering of hardmetal with microwaves leads to a finer microstructure because of lower sintering temperatures and shorter processing times compared to conventional sintering. It also reveals the potential of microwave sintering to provide a fundamental innovation in the powder metallurgy route of cemented carbides. A more recent sintering of premixed and prealloyed Cu-12Sn bronze for temperatures corresponding to transient, solid-state, and supersolidus sintering are done by Sethi, Upadhyaya and Agrawal (2003). The study proves bronze was microwave sintered in significantly less time as compared to conventional sintering. The hardness of the premixed microwave samples is higher than for the corresponding conventional premixed samples. In addition, the microstructure in the case of microwave sintered samples is more uniform than the conventionally sintered ones. They concluded their study with a recommendation to compact the premixed bronze at higher pressure, followed by microwave sintering for fabricating bearing and filters with higher porosity.

G. B. Kiat (2005) has worked on sintering tin base composites with 4% ammonium carbonate and another set of samples with 4% salicyclic acid in a microwave furnace. It follows the general prediction of the sintering properties for samples with pores forming additives. He has proved that as the sintering temperature increases, the density decreases while the porosity increases, both proportionally. He also sintered tin base composites without additives and found out that as the sintering temperature increases, the density increases while the porosity decreases, both proportionally.

The Fraunhofer IFAM Institute in Dresden, Germany is studying microwave sintering of PM ferrous and aluminum alloys and special materials to evaluate the potential for industrial scale production (http://www.metal-powder.net). It reports that lower sintering temperatures, shorter process time and improved material properties may result from effects created by the electro-magnetic field of microwaves. The work is conducted with the University of Bayreuth.

Tin was one of the first metals known to man. Throughout ancient history, various cultures recognized the virtues of tin in coatings, alloys and compounds, and use of the metal increased with advancing technology. Today, tin is an important metal in industry even though the annual tonnage used is much smaller than those of many other metals. One reason for the small tonnage is that, in most applications, only very small amounts of tin are used at a time (http://www.Key-to-Metals.com). Tin has a low melting point which is 232°C and therefore are to be sintered at low temperature. This enables using a modified domestic microwave furnace for this particular study.

There are four chosen parameters in this study which are sintering temperature, compaction pressure, type of sintering and sintering time. These parameters have been used in many research works. For example, Meng (2005) study the influence of sintering temperature on semi-conductivity and nonlinear electrical properties of TiO_2-based varistor ceramics. Based on the single sinter process, five disk samples of (Sr, Bi, Si, Ta)-doped TiO_2-based varistor ceramics, which were sintered separately at the temperature of 1200–1400°C for 2.5 hour were fabricated. The influence of sintering temperature on semi-conductivity and nonlinear electrical properties of these samples were investigated by measuring the properties of complex impedance, grain resistance, grain boundary resistance, breakdown voltage and nonlinear coefficient. Meng found that, between 1200 and 1400°C, breakdown voltage, nonlinear coefficient, and grain boundary resistance decreases gradually with decreasing sintering temperature, meanwhile grain resistance increases.

An example of a study of compaction pressure influence is done by Wilhelm et al (2001). He studied the influence of resin content (15-20, 25 w.t.%) and compaction pressure (75, 100, 125 MPa) on the mechanical properties and microstructure of SiC-Si composites with sub-micron SiC microstructure was evaluated. From his research, he found that the highest value of strength was observed at 733 MPa for the composites produced of SiC powder with a mean grain size of 0.70μm and a resin content of 25 w.t.%. Within this composite, the free silicon content was reduced to 12 vol.% and this is a reduction of more than 50% from previous experiments. These results are a consequence of the lowering of the free silicon content of the composites when increasing the resin amount. With decreasing SiC starting particle size the mechanical properties decreased but they were independent of compaction pressure.

Breval et al. (2005) did a research on the comparison between microwave and conventional sintering of WC/Co composites. WC/Co samples sintered in a microwave field differ radically in terms of phases, chemistry, and microstructure when compared with conventionally sintered samples. Microstructural investigations by TEM showed that in microwave sintered material the cobalt phase dissolved nearly no tungsten, whereas in conventionally sintered samples up to 20 wt.% tungsten was dissolved in the cobalt binder phase. Besides, smaller WC grains and finer and more uniform distribution of cobalt binder were observed in microwave sintered samples. This resulted in a harder material, which also exhibited better resistance towards both corrosion and erosion. Microwave sintered samples also have a three dimensional uniform shrinkage, whereas conventionally sintered samples showed a greater vertical shrinkage. According to his research, it is possible to microwave sinter WC/Co at lower surface temperature and in much shorter times than normally needed in a conventional furnace to obtain the same degree of densification.

In terms of sintering time, Upadhyaya (2001) has made advances in sintering of hard metals. His study emphasizes on the strategies to reduce the sintering time and improving properties in the processing of cemented carbides. He has shown that the cycle time for consolidating metal cutting grades can be reduced by as much as 70% by employing a fast dewaxing-rapid sintering approach. For mining and metal-forming grade hard metals, a thermal-cycling approach during sintering leads to a more homogeneous and less contiguous microstructure, which results in an enhanced toughness without compromising the wear resistance.

Literature review shows that there are some works on open porosity of sintered tin-copper-antimony and the effect of salicylic acid addition by Shahir (2003). Suzy studied the effect of ammonium carbonate addition on tin base alloys (2004). These two materials have proved to increase the open porosity of the alloy produced but reduce the overall density and hardness. In addition, some works has been done on sintering of tin base alloys in conventional furnace so as to produce self-lubricating bearings in base alloys. In the present study, the same experiment conditions are chosen as Yusof (2004). According to Yusof study, high compaction pressure, low sintering temperature and high sintering time is required to get high density of tin base alloys. For high open porosity, low compaction pressure, high sintering temperature and low sintering time is favorable.

From the literature survey, it has been observed that no microwave sintering work has been done on tin based alloys. Furthermore, no work has been done to optimize the input variables using Pareto ANOVA technique. There exists a great need for investigating the effect of microwave sintering of tin based alloy on density, porosity, dimensional changes, hardness and microstructure.