

CONSOLIDATION OF SINGLE AND DOUBLE LAYERS CERAMIC
STRUCTURE

NUR FARHANI BINTI ISMAIL

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Universiti Tun Hussein Onn Malaysia

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ABSTRACT

Basically, the performance of ceramic structure is essential for the development of ceramic system or component. This performance can be controlled from the early stage i.e preparation of raw material up till the final stage i.e. finishing steps. In this work, ceramic processing parameters at intermediate stage (consolidation) were considered to obtain good properties and structure of alumina ceramic. The microstructure, mechanical and physical properties of the green and sintered alumina ceramic pellets (single and double layer) were investigated. Some modification during consolidation (drying and firing) which focuses on the particle characteristics, drying techniques, additive and types of sintering were conducted. Three drying techniques (room, oven and microwave) were performed to the single and double ceramic alumina. Several factors which are identified as strong influence towards the sintered body i.e. characteristics (particle size and particle size distribution), MgO additive and sintering types (conventional and HIP technique) were selected as controlled parameters during the sintering step. All samples were fabricated using uniaxial press prior to the consolidation stage and were characterized using standard procedures. Based on the observation and measurement, it was found that the microwave drying technique produced a homogenous and uniform microstructure compared to the other drying techniques for both green and sintered bodies. The density value of 25 μm sintered body (Al_2O_3) with microwave drying technique shows an increment value from 3.551 g/cm^3 to 3.624 g/cm^3 compared to others drying technique. The same phenomena were also observed with the addition of MgO. In fact, the highest density value was obviously shown with alumina pellets sintered under HIP with same drying technique. In general, the increased of density value causes a reduction of porosity thus produce a good and hard sintered structure for all sintered samples.

ABSTRAK

Pada dasarnya, prestasi struktur seramik adalah penting untuk pembangunan komponen atau seramik sistem. Prestasi ini boleh dikawal dari peringkat awal iaitu penyediaan bahan mentah sehingga peringkat akhir iaitu langkah penyudahan. Dalam kajian ini, parameter pemprosesan seramik diperingkat pertengahan (pengukuhan) dipertimbangkan untuk mendapatkan sifat-sifat dan struktur alumina yang baik. Mikrostruktur, sifat fizikal dan mekanikal pelet alumina yang belum dan telah disinter (lapisan tunggal dan dua lapisan) telah disiasat. Beberapa pengubahsuaian semasa pengukuhan (pengeringan dan pembakaran) diberi tumpuan ke atas ciri-ciri zarah, teknik pengeringan, bahan penambah, dan jenis persinteran yang dijalankan. Tiga teknik pengeringan (bilik, oven dan mikrogelombang) telah dijalankan keatas seramik alumina tunggal dan dua lapisan. Beberapa faktor telah dikenalpasti mempunyai pengaruh yang kuat terhadap jasad yang telah disinter iaitu sifat-sifatnya (saiz zarah dan taburan saiz zarah), bahan penambah MgO dan jenis persinteran (teknik konvensional dan HIP) telah dipilih sebagai parameter kawalan semasa peringkat sinteran. Semua sampel telah dibentuk menggunakan tekanan satu paksi sebelum ke peringkat pengukuhan dan pencirian dilakukan mengikut langkah piawaian. Berdasarkan pemerhatian dan pengukuran yang dijalankan, didapati pengeringan menggunakan mikrogelombang menghasilkan mikrostruktur yang homogen dan seragam keatas jasad yang belum dan telah disinter berbanding dengan teknik pengeringan lain. Nilai ketumpatan bagi jasad $25 \mu\text{m}$ (Al_2O_3) yang dikeringkan dengan teknik mikrogelombang menunjukkan peningkatan dari 3.55 g/cm^3 kepada 3.624 g/cm^3 berbanding dengan teknik pengeringan yang lain. Fenomena yang sama juga dikenalpasti dengan bahan tambahan MgO. Secara umum, peningkatan nilai ketumpatan menyebabkan pengurangan keliangan maka akan menghasilkan struktur yang baik dan kuat untuk sampel yang disinter.

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LIST OF UNIT ABBREVIATIONS

Al ₂ O ₃	Aluminium Oxide
SiO ₂	Silicon Oxide
TiO ₂	Titanium Oxide
Li ₂ O ₃	Lithium Oxide
Y ₂ O ₃	Yttrium Oxide
ZrO ₂	Zirconium Oxide
MgO	Magnesium Oxide
°C	Degree Celcius
MW	Microwave
HIP	Hot Isostatic Press
XRD	X-Ray Diffraction
PSA	Particle Size Analyzer
TGA	Thermal Gravimetric Analysis
PVA	Polyvinyl Alcohol
EDS	Energy Dispersive Spectroscopy

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CHAPTER 1

INTRODUCTION

1.1 Research background

Generally, the production of ceramic component needs a sequence step which ranges from loose particles (powder) to the formation of hardened solid structure [3]. One of the most important stages in this ceramic processing is the consolidation step which consists of the drying and sintering stages [4, 5]. Controlling the processing parameters or raw material formulation is very crucial to ensure the ceramic properties and structure is in good condition. Basically, the drying process of the ceramic materials is very crucial since it influences the physical, mechanical and characteristics of the ceramic body [6]. Basically, the drying process is a complex process involving simultaneous heat and mass transfer that will contribute to the loss of moisture from the pore structure [7]. Theoretically, the movement of moisture (in a ceramic body) is strongly related to the shrinkage mechanism, and this is a major defect in ceramic product where this defect can range from the initiation of crack to the failure of the fired body due to the crack propagation [8]

The next stage of ceramic consolidation step is sintering. Most dried ceramic products must go through the sintering process in order to produce the dense product with desired properties such as low porosity and high strength. Typically, the sintering stage is the most critical step in ceramic processing because it involves the complex mechanism in diffusion i.e., diffusion, mass transfer and grain growth etc. Sometimes, the sintering aid (additive) is used during the sintering process to promote sintering or controlling of the grain growth in producing good ceramic structure [9, 10]. Furthermore, the sintering aids are often used in ceramic processing in order to promote faster consolidation at low temperature especially for pure

alumina. Basically, pure alumina must be fired at high temperature in the range of 0.5 until 0.75 of the melting point [4] and it takes a long time to achieve high density. Densification of the ceramic structure is not only influenced by the impurities (dopant) but also influenced by the characteristic of the materials itself (i.e. particle size, particle size distribution, particle shape, particle aggregate). One of the powder characteristics is particle size distribution of the raw materials. Basically, narrow particle size distribution promotes more densification [11, 12] as compared to broader particle size. Besides that, the use of the pressure during sintering also can help increase the densification rate. One of the most common methods used is HIP where the pressure and inert gas were supplied during the process. This technique has potential to produce high density with fine grain size for the pure ceramic.

As elaborated in detail in the previous paragraph, there are many factors that influence ceramic sintered properties and structure. Most of the influence factors are in consolidation (drying and firing). Therefore, in this investigation and observation of these critical parameters or factors to final structure were considered.

The main emphasis of this study is to investigate the effect of drying heating (room, oven and microwave), particle size (in range below 90 μm) and MgO additive to the densification and microstructure of the Al_2O_3 pellet. The drying heating is also investigated on the double layer pellet ($\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3 / \text{SiO}_2$) for better understanding about the microstructure evolution. Analysis by utilizing scanning electron microscope (SEM), Energy Dispersive X-Ray Spectroscopy (EDS) and X-Ray Diffraction (XRD) were carried out to investigate the microstructure, formation of the SiO_2 and MgO and phase change. The characterisation of the physical (porosity, density and shrinkage) and mechanical properties (hardness) also were performed because these are important properties for the ceramic structure.

1.2 Problem statement

Fabrication of the ceramic structure is very complicated because it involves water removal and diffusion mechanism during the drying and firing (consolidation). In general, properties and microstructure i.e. density, porosity, hardness and shrinkage are strongly influenced by the drying and firing (consolidation) process. There are many factors that influence the dried and sintered ceramic body i.e. powder characteristics, drying technique, sintering technique and sintering aid. However, the

problem still arises when trying to develop ceramic structure during drying and sintering, for example the densification is not easy to achieve for the ceramic body structure, especially for hard Al_2O_3 ceramic particles. Therefore, further investigation and observations need to be conducted to examine the effect of processing parameters and factors to the consolidation process.

1.3 Aim

The main purpose of this study is to investigate the effect of the consolidation stage (drying and sintering) with the influence of factors (powder characteristics, sintering aid and sintering condition) towards properties, microstructure and morphology of ceramic structure.

1.4 Objectives of study

The objectives of this study are:

- i. To determine the effect of different drying to the Al_2O_3 ceramic structure (slurry and pellet type).
- ii. To determine the effect of different drying techniques towards the sintering structure (single and double layer).
- iii. To develop the sintered single Al_2O_3 structure via conventional and HIP technique with controlled variable factors (particle size and additive)

1.5 Research Scopes

An understanding of the properties of the ceramic structure will lead to a deeper understanding on the impact of its performance on the procedures that need to be considered to develop good ceramic single and double layer structure. Therefore, the scopes of this study have been outlined as follows:

- i. Development of shell mould green body (slurry type) by using three drying technique (room, oven and microwave).

- ii. Preparation of the single Al_2O_3 (with and without MgO additive) structure and double Al_2O_3 layer ($\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{SiO}_2$) ceramic structures from different particle sizes by powder (uniaxial) pressing.
- iii. Development of single Al_2O_3 structures by using three drying techniques (room, oven and microwave) and two sintering techniques (conventional and HIP).
- iv. Development of double Al_2O_3 layer structures by using three drying techniques (room, oven and microwave).
- v. Characterization of the phase analysis of Al_2O_3 , SiO_2 and MgO powder before and after the consolidation process by using X-ray Diffraction Analysis (XRD).
- vi. Observation of the microstructure of Al_2O_3 single and double layer structures by using Scanning Electron Microscope (SEM).
- vii. Characterization of the physical properties testing (porosity, density and shrinkage) and mechanical properties (hardness) for Al_2O_3 structure.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of ceramic materials

Ceramic materials are inorganic, nonmetallic materials which compose at least two elements. Most of the ceramics are compounds between metallic and nonmetallic elements which is bonded together primarily by ionic and/or covalent bonds. Basically, most of the ceramic materials must go through a heat treatment process (firing) at high temperature to produce the materials with desired properties. This phenomenon is strongly related to the term of “ceramic” which comes from the Greek word *keramikos*, which means “burnt stuff [13].

Ceramics can be divided into two groups, which is traditional ceramic and advanced ceramics. Traditional ceramics include silicates such as clay, pottery, bricks, porcelain and this group is still implemented until now in the industry as a refractory due to high temperature resistance. Advanced ceramics is development of the new ceramic product which have high temperature resistance, chemical, mechanical characteristics and electrical goods in advanced technology. Basically, advanced ceramic materials consist oxides such as alumina (Al_2O_3), silica (SiO_2), zirconia (ZrO_2) and barium titanate (BaTiO_3) and non oxide group such as silicon nitride (Si_3N_4), boron nitride (BN) and carbides [4].

Recently, ceramic materials have been widely used in the industry especially in advanced technology such as refractories, spark plugs, dielectrics in capacitors, sensors, abrasives, and magnetic recording media [4]. Most of the ceramic materials are generally porous materials, brittle, high temperature and highly sensitive to the physical thermal changes. Basically, properties of ceramic products are strongly related to their chemical compositions and their atomic and micro scale structure [1]. Thus, controlling the materials and processing operation is needed to produce

product with good performance. Ceramic materials originally from powders and must go through fabrication process to produce product.

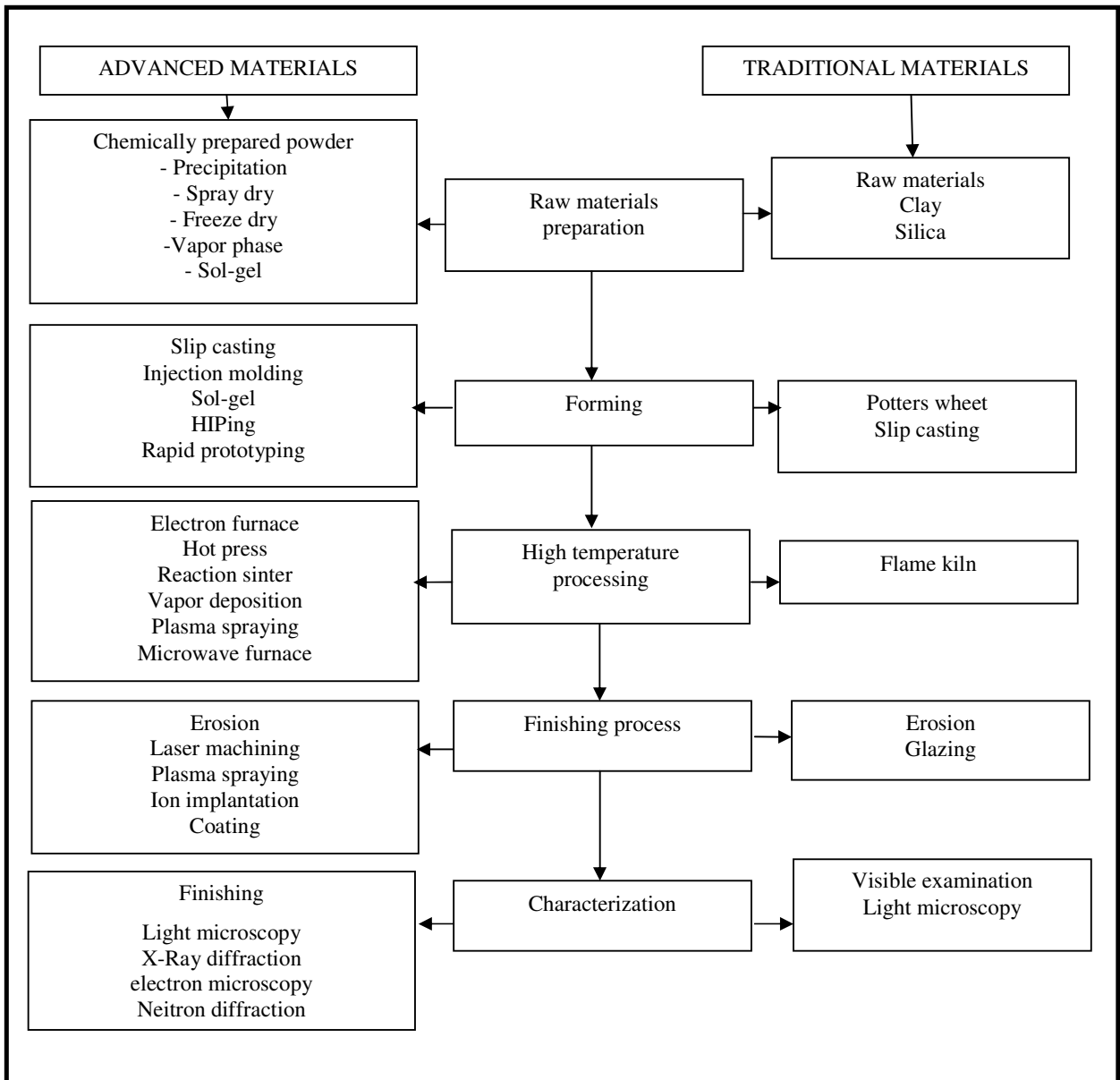


Figure 2.1: A comparison of different aspects of traditional and advanced ceramics [14]

2.1.1 Alumina (Al_2O_3)

Alumina is known as an alumina oxide or aluminum oxide which has chemical formula Al_2O_3 . Generally, alumina is formed by metal aluminium and occurs naturally as the mineral corundum (Al_2O_3), diaspore ($\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$), gibbsite ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) and most commonly as bauxite which is impure form of gibbsite. Alumina also known as many other names such alpha, gamma and delta regarding to its nature and industry [15, 16]. Alumina is produced by using Bayer Process which extracting alumina from bauxite.

The basic unit cell of aluminium oxide has two types of sites which are hexagonal and octahedral. Hexagonal sites are the corner atom in the cell while the octahedral sites are present between two layers of vertical stacking. Aluminium cations are in $2/3$ of the octahedral sites and oxygen anions are in $1/3$ of the octahedral sites. Each oxygen is shared between four octahedral sites permit strong bonding and therefore give rise to the characterizations of the properties of alumina [17].

Alumina is commonly available and is graded in different purities and it typically graded into two main groups. The first of high alumina grades with at least 99 % and the second alumina grades between 85% purity and going up to 99.9% Al_2O_3 [18, 19]. These main groups can be further divided into subclasses according to type, purity and intended services [20]. Typically, the selection of material composition (purity) is important because each composition has a different percent alumina content and influence of material properties. Basically, alumina with high purity has the best mechanical properties with high density ($>3.75\text{g}/\text{cm}^3$) and high sintering temperature ($1500^\circ\text{C} - 1900^\circ\text{C}$) [20].

Generally, alumina is used either in pure form or as raw material mixed with other oxide [4] and typically used as a base materials due to the high strength. Alumina is the most cost effective and widely used materials in family of engineering ceramics. The raw materials with high performance technical grade ceramic is made readily available and reasonably priced, resulting in good value for the cost fabricated alumina shapes. Alumina has unique combinations and useful for electrical, mechanical and physicals properties [15]. Alumina is widely used for engineering application such as ceramic, refractory, chemical industry, catalyst, filler and glass industries due to its abundance and its multiple form as well as its

properties. The most and wide ranging use of alumina in the field of ceramics as an insulating materials, electronic and mechanical ceramics[16].

Basically, the physical properties such as density, porosity and shrinkage are important criteria that should be considered in the material selection process. Good physical properties of the materials are very important to ensure that the material can be used with optimum [3]. Mechanically, the alumina ceramic has great properties in hardness, resistance to abrasive wear and dimensional stability. Basically, the strength (hardness) properties of sintered alumina are strongly influenced by the microstructure; porosity, grain size and pore size.

The excellent combination of mechanical and electrical properties such as wear resistance and good hard makes extensive usage in the manufacturing industry [15]. There are many ceramic processing methods can be used to produce a product of the alumina with various sizes and shapes. Alumina can also be combined with materials other metal or ceramic material such small amounts of silica, magnesia and zirconia to obtain the desired properties suitable for the application.

2.1.2 Silica (SiO₂)

The compound silica (SiO₂) is typically formed from silicon and oxygen atoms [21]. A chemical compound is defined as a distinct and pure substance formed by the union of two or more elements. Silica (SiO₂) or silica dioxide is the most simple silicate materials and it is obtained by in minerals, such as quartz and flint, and in plants such as bamboo, rice and barley.

The three major forms of crystalline silica -quartz, tridymite and cristobalite- are stable at different temperatures In its natural form it mostly occurs as a crystalline phase and rarely in an amorphous state. Various phases may be formed, depending on temperature, pressure and degree of hydration. At atmospheric pressure the anhydrous crystalline silica may be classified to some phases according to the temperature.

Silica is a hard, chemically inert and has a high melting point attribute to the strength of the bond between the atoms. Basically, the silica widely used in industrial development especially in glass, foundry and ceramic depending on their characteristics. It is widely used because it is inexpensive, hard, chemically stable and relatively infusible and has the ability to form glasses.

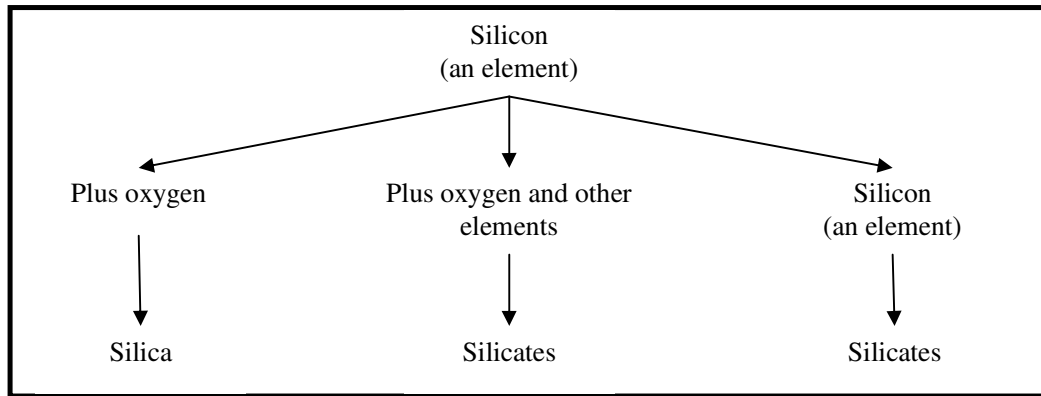


Figure 2.2: Relationships among the chemical term on silicon [22]

2.2 Fabrication of ceramic component

Basically, fabrication of ceramic component involves several stages which starts from the formulation of the raw materials in the powder forms or particles till the characterization of solid end products as shown in Figure 2.3.

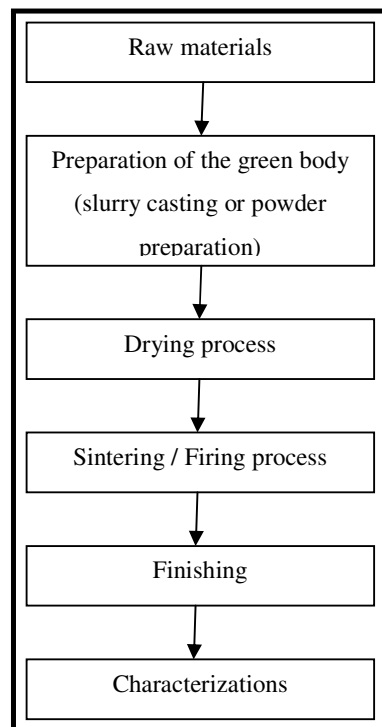


Figure 2.3: Processing of the ceramic structure [3]

Typically, ceramic or particle powder is used as a basic in fabrication of the ceramic body for producing green body and sintered body structure. This ceramic powder or particles need to be characterized prior to the next processing stage of the ceramic [4, 5, 23]. This is because starting powder always strongly influences the ceramic green body as well as to the sintered body. Therefore, the characterizations of the ceramic powder become greatest interest in ceramic formulation i.e. particle size, powder agglomerate and exaggerated [4, 24, 25]. Previous studies in this field of characteristic properties show that both of the particle distribution either narrow or broader promote the densification rate at the low sintering temperature [12, 25-28]. In fact, most ceramic investigation consider particle geometry [24, 25] and behaviour [4, 5] relates them to the end product of ceramic properties.

The next stage of the processing ceramic route is the forming stage where the raw material of the ceramic is produced to desired size or shape before the densification process. Usually, this can be done using several techniques such as compaction, casting, extrusion and many more [3, 4, 15]. Compaction or pressing is one of the common techniques that is commonly used to produce the green part of ceramic. In this work, the pressing technique is applied in the fabrication of ceramic alumina powder. The pressing technique is a process of compaction and shaping of a dry and semidry powder or granule material in a rigid die or flexible by applying sufficient pressure directly in vertical direction. In general, compaction (pressing) processing consists of the three steps: filling of the die (mould), powder compaction and ejection of the compacted powder and this technique is widely used in ceramic processing due to the simple formation technique and gives accurate dimension [3]. However, some of the ceramic powder is difficult to compact especially for the very fine and hard particles. Due to this condition, a small binder is needed during this forming process to create a sticky surface so that particle can bind easily in producing green body [4, 29]. For the example, polyvinyl alcohol (PVA) is commonly used as a binder for ceramic oxides (as shown in Table 2.1) and also can act as the lubricant of the ceramic compaction and it is always used to remove the compacted green body from the mould. Furthermore, the lubricant is also important in reducing distortion [30].

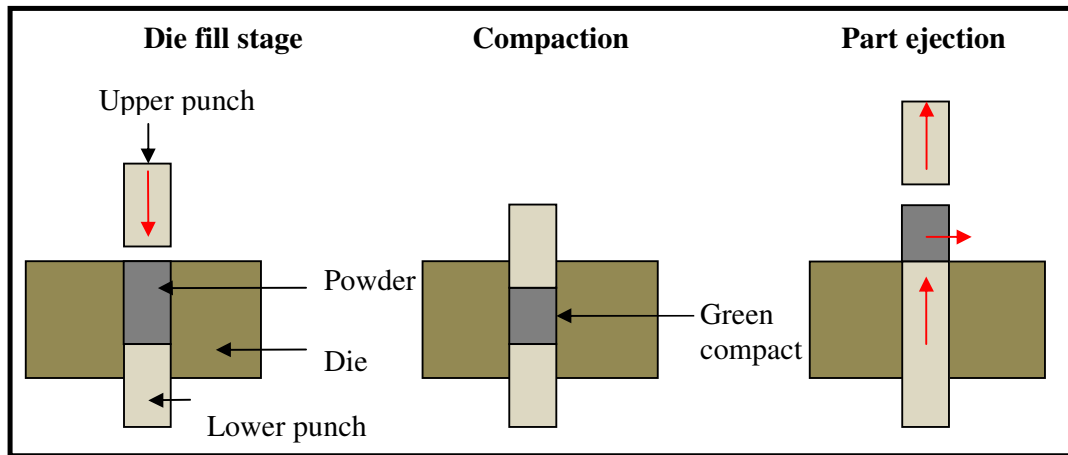


Figure 2.4: Schematic for powder compaction process [3]

Table 2.1: Additives used in Powder Pressing

Ceramic powder	Additive		
	Binder	Plasticizer	Lubricant
Alumina	Polyvinyl alcohol	Polyethylene glycol	Aluminiumstrearate
MnZn ferrite	Polyvinyl alcohol	Polyethylene glycol	Zinc strearate
Barium titanate	Polyvinyl alcohol	Polyethylene glycol	Stearic acid
Alumina substrate / spark plasma	Microcrystalline wax emulsion	KOH : Tatnic acid	Wax, talc and clay
Steatite insulator	Microcrystalline wax and clay	Water	Colloidal talc and wax
Refractories	Ca /Na lingo sulfonate	Water	Stearic acid
Tile	Clay	Water	Colloidal talc and clay

Then, the next stage of ceramic processing that is considered is the consolidation stage. This is because the processing of ceramic components used powder as particles that need to be dried and fired to form a hard and strong body. Because the ceramic has properties of high melting temperature, inert and highly sensitive to thermal physical changes. Therefore, this ceramic component needs to be done in a controlled environment for producing good and quality of the end cast products. In fact, there are many factors or parameters that can influence this consolidation stage [24] which directly will influence the ceramic property. This will be explained in detail in the following section.

2.3 Drying on ceramic structure

In consolidation of ceramic powder to the green ceramic component, the drying needs to be carried out prior to the next sintering stage. This process plays an important role in ceramic forming operation to ensure the quality of structure is in good condition. Basically, drying needs to be done before heating (firing) to make sure free water is removed and avoid abrupt thermal changing which can cause steam spallation occur to higher sintering temperature.

In ceramic processing, drying is one of the most important processes especially for the ceramic porous material where the pore is filled with moisture or liquid [8, 31]. Therefore, the drying needs to be conducted in order to remove free water from the green body which brings the drastic changes in thermal gradient during this stage and at the same time effects the sintered body structure at the final stage of the processing. Drying of the ceramic materials involves complicated mechanisms because normally it will exhibit the shrinkage. Therefore, most of the ceramic must be dried as thoroughly as possible slowly and carefully to avoid failure during the drying process [32].

2.3.1 Mechanism of drying in ceramic materials

Basically, drying is a process to remove moisture from the body by applying the heat [33]. The drying process in most ceramic materials is very crucial since it can influence the mechanical, physical and characteristic of the body. Typically, this process is accompanied by physical and structural changes that are called shrinkage mechanisms.

In drying, there are several physical mechanisms which contribute to moisture migration such as moisture transport either by individual diffusion and capillary flow or combined effect of moisture, temperature or pressure gradient [33]. In addition, drying materials are classified into hygroscopic and non hygroscopic materials. For the hygroscopic materials, there are two types of water found in porous materials like ceramic which are free water and bound water. Free water is water filled in ceramic cell cavity and it is easy to evaporate while bound water is referring to the water in a ceramic bonded to wall cell [7]. Typically, the bound water

is chemically attached to the cell wall and needs a large amount of energy to evaporate.

Drying mechanisms involve the evaporation and migration of phase (liquid or gas) from interior part to surface and this is a continuous process until to the body is dried as shown in Figure 2.5. The drying consists of two parts; linear and non linear. For the linear, water moves from the internal to the surface which involves the capillary suction (free water) and non linear, the moisture is driven out from the pore by diffusion (vapour phase) mechanism. All these movement and phase migration relates to the heat, mass and gas transport between interior dried body and external environment. This mechanism of water always depends on the hygroscopic nature (water held to the particle) and properties of the materials, drying condition and the way heat is applied (external or volumetric heating). As mentioned in the paragraph above, two types of water found in ceramic and free water is usually evaporated first as compared to the bound water at the beginning of the drying process. The free water is fully removed during the drying process and it is slightly different to the bound water which is removed by applying a large amount of heat.

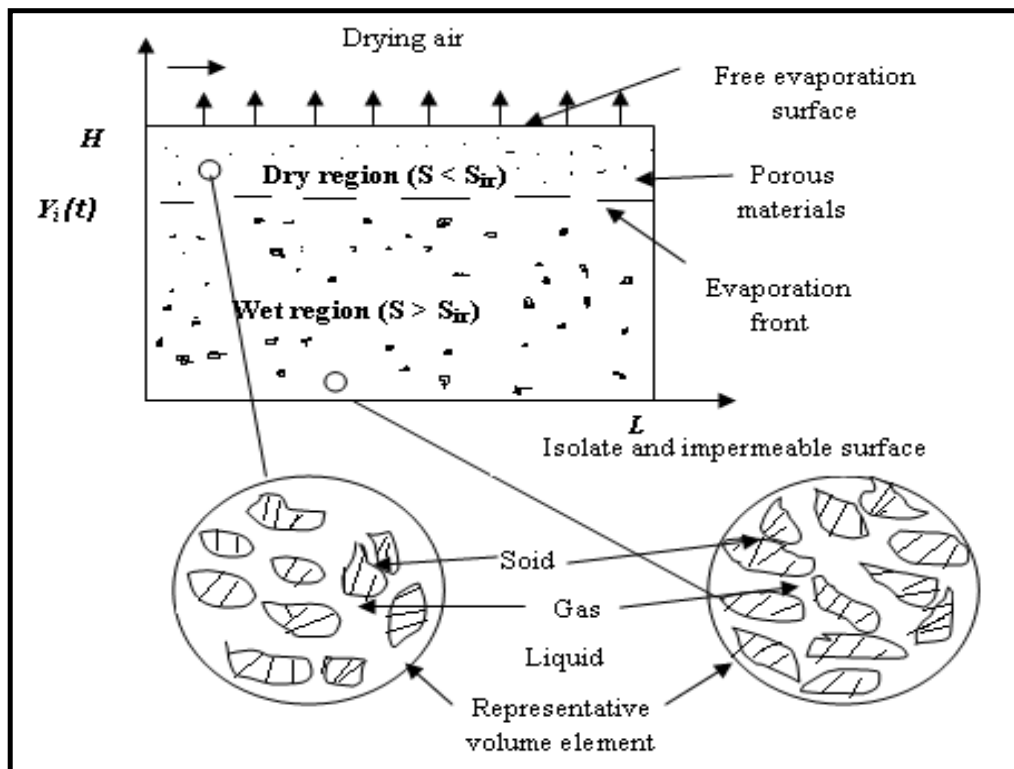


Figure 2.5: Schematic of the drying model [7]

2.3.2 Drying technique

There are several methods of drying which are commonly used to remove water or moisture such as conventional (room and oven) microwave and etc., every method has their own characteristics and techniques to dry body structure (green body) and this indirectly influences the final structure.

2.3.2.1 Room drying

Room drying is one of the most common drying methods and it is a traditional method that is still widely used up till now. This procedure of drying strongly depends on the outside environment [34]. In addition, drying is frequently a dual process which involves evaporation and migration. Theoretically, conventional drying involves the transportation of heat to the surface of material and then followed by the movement of water from the heated body. However, in room drying, the evaporation only acts immediately to the surface of the body and delayed action in the migration of water from the internal structure (heated body) which is dependent on the rate of diffusion through the materials [32-34]. One advantage of room drying is low initial cost. However, there are many disadvantages for this method; low energy efficiency and more time consuming heating processes especially for thick materials [35, 36] to remove water from the heated body during the migration process. In general, room drying is typically controlled by the condition of the environment where it is difficult to predict the drying quality and speed of air. Therefore, this condition can strongly give influence to the excess of the moisture movement that will result to uncontrolled shrinkage mechanism. Furthermore, this shrinkage mechanism gives high impact which leads to the crack formation and finally brings to the failure of materials. Thus, imperfect heating can cause product rejection and energy waste due to the uncontrolled parameter of the drying system. Therefore, determining of the proper drying technique is needed in order to minimize the microstructure defect and the processing time cycle.

2.3.2.2 Oven drying

Oven technique is a common laboratory drying method and is one of the alternatives to drying that can reduce the processing time and cost of production. In general, proper methods which can control the temperature is essential for quality of the ceramic structure. This is because ceramic becomes weak when drying occurs rapidly at high temperature. However, this drying technique is slightly different than the room condition due to its potential to control every processing parameter by setting the soaking heating time and also heating rate at the beginning stage [34]. Therefore, among the advantages of this drying is that it can dry products successfully, and at the same time it can help can avoid the failure of the ceramic body. Basically, the slow changes of temperature gradient will initiate the internal diffusion process of pore water that brings to the slow motion of water to the dried boundary layer. This mechanism needs a slow and controlled environment especially for the pore section of brittle materials that are highly exposed to the shrinkage problem. However, this drying technique also has its disadvantages where the heat only acts on the surface than the internal will result in the removal of the surface shrinks than the bottom. Furthermore, the oven is not an efficient drying technique and also uses high energy to remove water from the internal structure during the period of the drying process.

2.3.2.3 Microwave drying

Today, the microwave technique is one of the most widely used in drying method approaches to improve the physical property materials because this method offers much greater production outcomes and offers various heating conditions [37, 38]. Basically, microwave drying is capable to create higher drying rate and energy efficiency than conventional heated air drying. Several works related to this microwave drying technique mentioned that microwave is widely applied to various kinds of raw materials and products including food, vegetables, fruits and wood as well as ceramic materials [32, 39]. In fact, microwave is one of the most clean drying technology as the usage of microwave heating can minimize the environment emission problems via its internal and volumetric heating mechanism, and at the same time can still produce better product quality [40]. Furthermore, this technique

can reduce the processing time because it allows the penetration of heat directly through the internal of material structure where the volumetric heating drives the water away from the interior, hotter section [41] and this technique is very important for most of the ceramic body to remove water completely (as shown in Figure 2.6). The drying process is important in ceramic body in order to remove water as much as possible for preparing samples before firing at high temperature techniques [8]. Due to the condition where heat can be generated throughout the volume of the materials and resulting volumetric heating, the possibility in getting rapid heating and uniform structure is high [8]. During microwave processes, the water in the material absorbs microwaves throughout the entire mass causing molecular vibrations with respect to the oscillating electric field of microwaves and thus heating simultaneously throughout the material as indicated [38].

Basically, the microwave heats materials internally within the materials instead of originating from external heating sources and the depth of penetration of the energy vary in different materials. During microwave processing, the microwave energy penetrates through the materials and some of the energy is absorbed by the materials and is converted to heat which can lead the interior part of the materials to become hotter than its surface due to the interior part achieving higher temperature and drying first; at the same time, it can reduce thermal stresses that cause cracking during processing [41]. In addition, this internal heating mechanism also offers a uniform energy absorption, heat and moisture distribution within the porous structure such as ceramic [39].

Microwave processing of materials offers the potential for reducing production time for ceramic materials due to the energy saving; and microwave energy has proven to be an efficient and reliable form of heating for a wide range of industrial processes [42]. Schroeder and Hackett [43] reported that a benefit from using microwave heating in foundry in which the process time is reduced to one half to one tenth of that required by conventional heating [37]. Therefore, microwave is an appropriate heating device including the potential for reductions in manufacturing costs and shorter processing time [44]. Table 2.2 shows the comparison of energy saving of ceramic manufactured by the conventional drying and firing, and microwave drying and firing. In contrast to conventional drying, heat generated by microwave energy depends on microwave absorption efficiency of materials including types and the dielectric properties of materials [45].

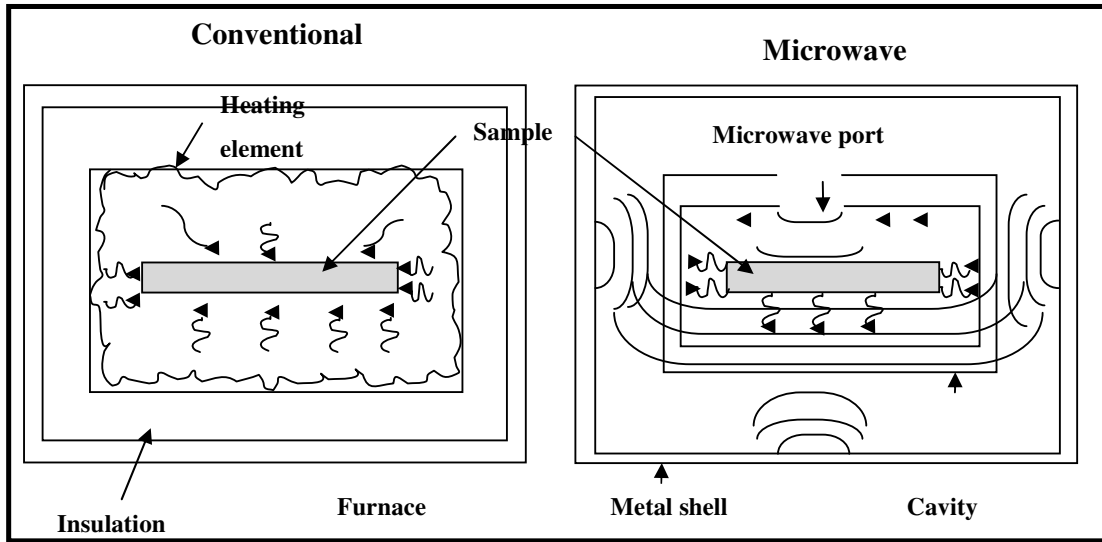


Figure 2.6: Heating pattern in conventional and microwave furnaces [41]

Table 2.2 : Comparison of energy savings for conventional and microwave drying and firing of ceramics [41]

Energy savings ($\times 10^6$ kW -h/yr)					
	conventional drying	microwave drying	conventional firing	microwave firing	Total energy saving
Brick and tile	56.1	28.05	198.9	19.9	207.06
Electrical porcelain	3.52	1.76	12.48	1.25	12.99
Glaze	16.63	8.3	58.97	5.89	61.37
Pottery	1.96	0.98	6.94	0.69	7.23
Refractories	10.87	5.4	38.53	3.85	40.08
Sanitary ware	25.04	12.52	88.76	8.88	92.4
Advanced ceramics	1.3	0.65	4.6	0.46	4.79
Total					
($\times 10^6$ kW -h/yr)	115.42	57.66	409.18	40.92	425.92
($\times 10^6$ PJ/yr)	0.42	0.21	1.47	0.15	1.5

2.4 Sintering of ceramic structure

Most of the unfired dried ceramic bodies must go through the sintering process to produce a hard and strong body by creating diffused particles and this is important for the ceramic production [46]. Actually, sintering is the process to convert powder to a dense product by bonding together the particles until it is adhere of each other and this process is well known as the thermally activated process of the compact. Generally, this process can be carried out at the temperature below the melting point which ranges from 0.5 to 0.75 of the melting point [4] until full densification is obtained. In fact, most of the ceramic bodies must be sintered to produce microstructure with the required properties. Basically, sintering is a process where the fully densification takes place and involves the development of new ceramic structure [4]. Typically, sintering also influences the characteristic of the ceramic structure and properties. This condition leads to the changes of ceramic performance such as strength, shrinkage, porosity, grain size etc. The strength of ceramic structure is increased due to the formation of diffused bonding between the particles [29]. This will also lead to the formation of grain growth; sometimes the abnormal grain growth. Typically, this complex process of sintering is influenced by a variety of parameters and factors such as powder characteristics (particle size), distribution of dopant addition and sintering conditions (temperature, applied pressure, time and atmosphere). At the same time, the shrinkage mechanism takes place in the sintered structure as the void spaces between the particles reduce when water starts to eliminate. Another characteristic is increment of average grain size. Sintering is divided into two basically categories: solid state sintering and liquid phase sintering.

Table 2.3: Selected ceramic composition and the sintering process used during densification [29]

Composition	Sintering process
Al ₂ O ₃	Solid state sintering with MgO additive Liquid phase sintering with silicates phase
MgO	Liquid phase sintering with silicates phase
Si ₃ N ₄	Liquid phase sintering with oxide additive (e.g Al ₂ O ₃ and Y ₂ O ₃) under nitrogen gas pressure or under an externally applied pressure
SiC	Solid state sintering with B and C additive Liquid phase sintering with Al, B and C or oxide additive
ZnO	Liquid phase sintering with B ₂ O ₃ and other oxide additive
BaTiO ₃	Liquid phase sintering with TiO ₂ rich liquid
Pb(Zr,Ti)O ₃ (PZT), (Pb,La)(Zr,T)O ₃ (PLZT)	Sintering with a lead rich liquid phase: hot pressing
ZrO ₃ / (3-10 mol %)	Solid state sintering
Mn-Zn and Ni-Zn ferrites	Solid state sinterin under controlled oxygen atmosphere
Porcelain	Vitrification
SiO ₂ gel	Viscous sintering

2.4.1 Solid state sintering

In general, solid state sintering consists of three stages (as shown in Figure 2.7) which is initial, intermediate and final [4, 5]. At the initial stage, the particles are in contact with each other and developed the neck growth by diffusion mechanism. Then, it is followed by the intermediate stage where pores shrink through the mass transport by the neck growth. Once the grain growth begins, the pore phases are intersected by grain boundaries and at the same time the microstructure can be developed at the final stage. Basically, the pores shrink continuously and cause small grains to be consolidated to form larger grains.

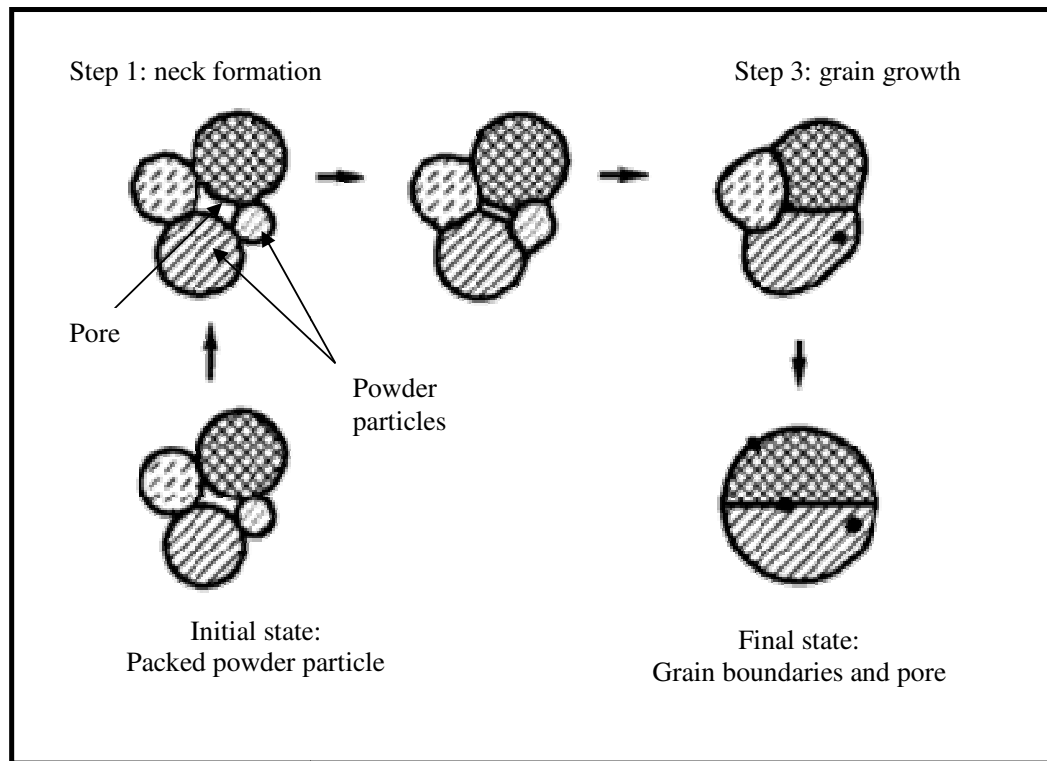


Figure 2.7: Schematic of three stages in sintering [4]

Sintering is typically accompanied by an increment of the free energy of a system. The driving forces for sintering (solid state sintering) which consists of three portions such as the curvature of the particle surface, an externally applied pressure and chemical reaction gives rise to the free energy with reduction of surface energy (as shown in Figure 2.8)

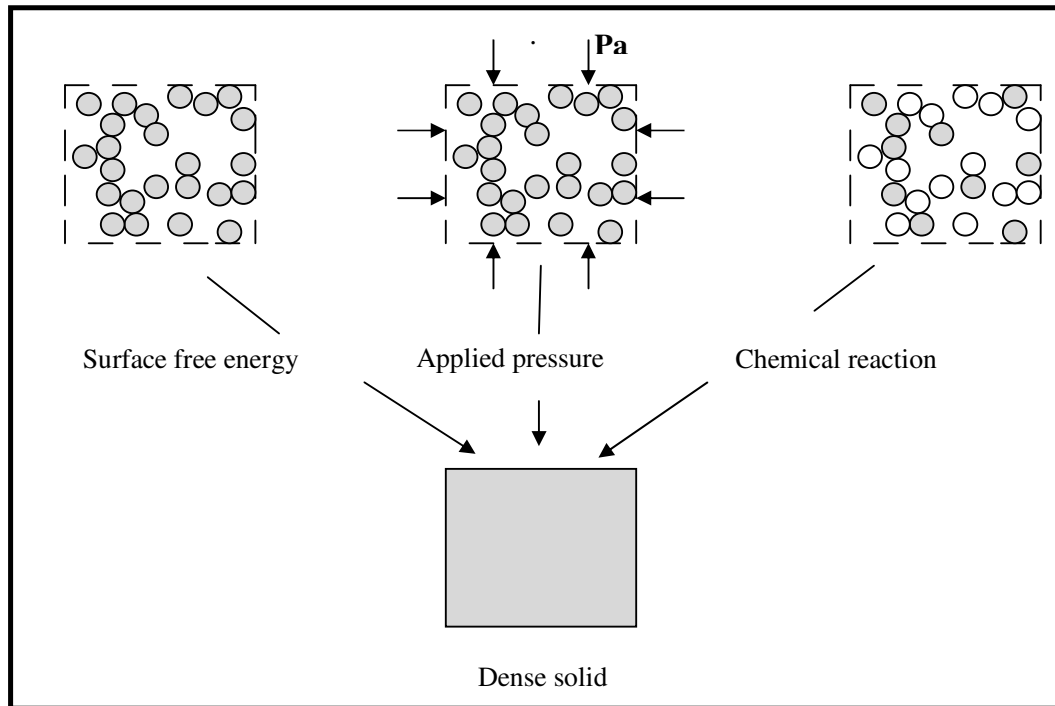


Figure 2.8: Schematic of driving force for sintering [4].

In general, the reduction of the surface free energy is accompanied by the densification process with producing shrinkage. This is due to the transport matter from inside the grain into the pores and another process is coarsening where there is no in producing the shrinkage [4]. Coarsening is a process of the rearrangement of matter between different parts of the pore surface to produce the microstructure and this process occurs simultaneously with the densification process, as shown in Figure 2.9. Basically, there are two characteristics of the solid state sintering which are shrinkage component and porosity [4].

In general, the common difficulty in densification for the polycrystalline (Al_2O_3) powder compact is it needs a high temperature during the sintering process and at the same time increases the sintering rate. However, the excessive sintering tends to cause the ceramic materials to become weak because of the porosity and grain growth. This is because the coarsening process is strongly related to the grain growth. The domination of coarsening phenomenon in solid state sintering typically will hinder the production of dense body with high density. Also, the coarsening process will promote an uncontrolled grain growth which is typically divided into

two categories: normal and abnormal grain growth. Thus, controlling the grain growth during the sintering process is one of the most important parts in the formation of good ceramic structure because uncontrolled grain growth may lead to the production of ceramic with undesirable properties such as low in strength and cracking. The sintering aid was introduced to the ceramic system in order to enhance the diffusion properties of the particles or improve diffusion by creating a small amount of liquid phase during sintering. In addition, controlling the starting powder is also needed in solid state sintering as the smaller particle size can increase the sintering rate. Therefore, controlling powder is important in regard to reduce sintering temperature and at the same time to produce the good properties of the ceramic body [4, 24, 25].

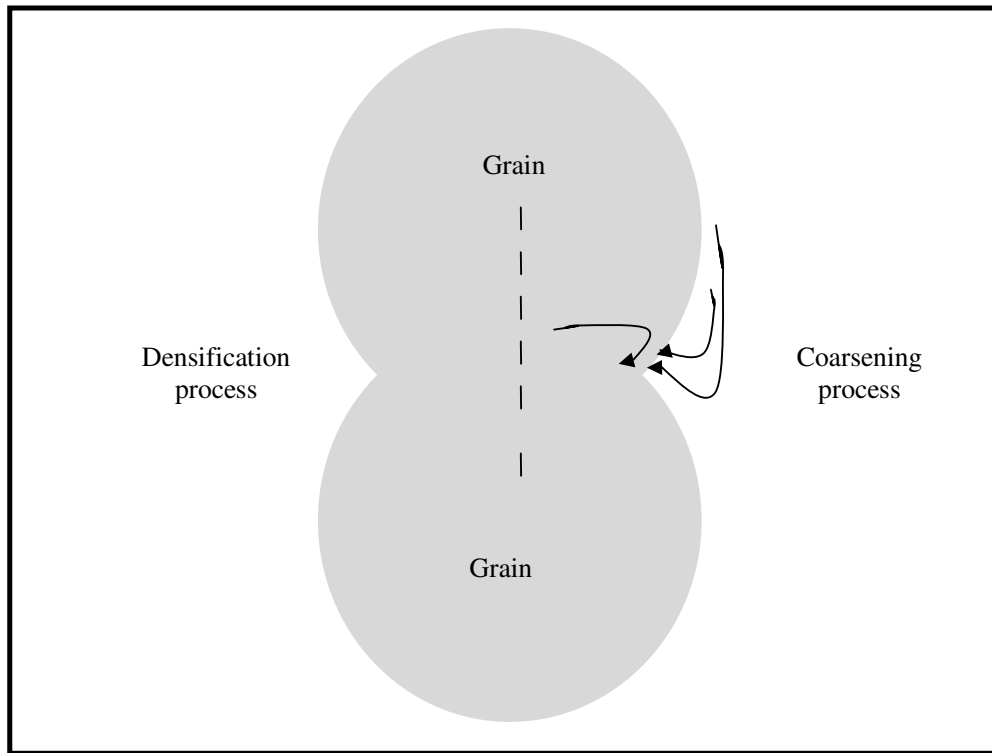


Figure 2.9: Schematic of densification and coarsening process of microstructure during firing process [4]

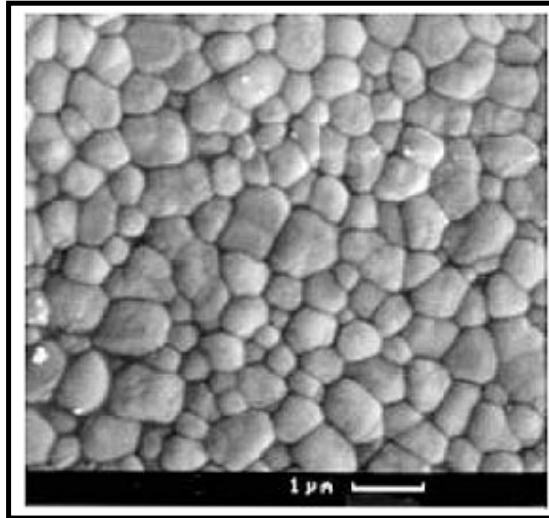


Figure 2.10: The surface of an Al₂O₃ ceramic during sintering [4, 29]

2.4.2 Liquid phase sintering

Another common process for densification of ceramic is sintering in the presence of the reactive liquid (liquid phase sintering). This process is always used for ceramic materials which are difficult to densify by solid state sintering in order to enhance densification rate, achieve grain growth or produce specific grain boundary properties. Furthermore, the liquid phase sintering is an effective densification process to alumina ceramic by adding an additive which can reduce the sintering temperature and processing time. Basically, the process of liquid phase sintering has three sequential stages which are particle rearrangement, solution precipitation and coalescence (as illustrated in Figure 2.11)[4].

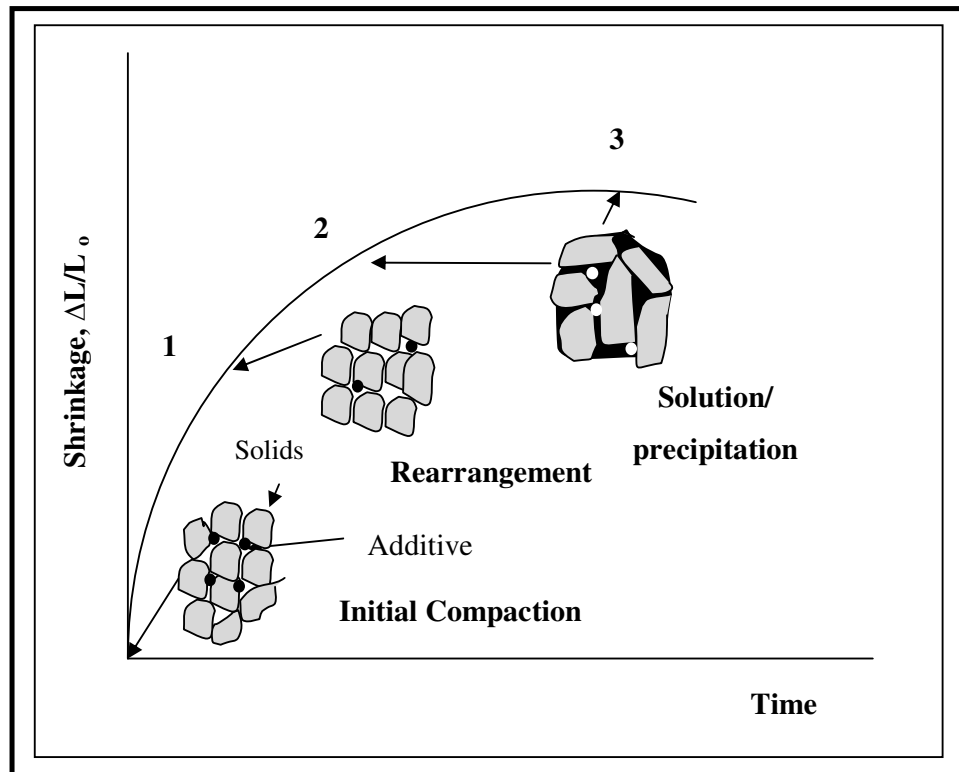


Figure 2.11: Schematic evolution of powder compaction during liquid phase sintering [4]

In the first stage, the liquid phase is formed with the particulate solid at the sintering temperature. Basically, wetting liquid plays an important role to provide a capillary force which can pull the solid particles together. Also, it can induce particle rearrangement due to the influence of surface tension, giving more efficient packing particle. This rearrangement of the particles can lead to faster densification with full density being achieved in a liquid phase sintering compared to the solid state sintering. Then, the densification proceeds to the next stage by a solid solution of the solid materials at the contact point, diffusion through the liquid phase and the precipitation at solid surface sites outside the contact area. Therefore, densification occurs at a faster rate in liquid phase sintering due to the materials being transported more rapidly in liquid than in solid. At the final stage of the liquid phase sintering is solid state skeleton network. This process is slow because there are large diffusion distances in the coarsening structure.

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