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**Ministry of Higher Education and Research** 

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# Investigation the effect of adding MgO nano size on properties of SiO2- Al2O3 system

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# الشكر والتقدير

لابد لنا ونحن نخطو خطواتنا الأخيرة في الحياة الدراسية من وقفه نعود بها الى اعوام قضيناها في رحاب جامعة بابل \ كليه هندسة المواد \قسم هندسة السيراميك ومواد البناء مع أستاذتنا الكرام الذين قدموا لنا الكثير باذلين بذألك جهوداً كبيرة في بناء جيل الغد لتعش الامة من جديد شكر مقدم الى مختبرات كلية هندسه المواد أقسم السيراميك لتسهيلهم متطلبات المشروع وقبل ان نمضي نقدم أسمى آيات الشكر والامتنان والتقدير والمحبة الى الذين حملوا اقدس رسالة في الحياة

> الى جميع اساتذتنا الافاضل . واخص بالتقدير والشكر استاذي المشرف : الاستاذ المساعد الدكتورة الهام عبد الماجد



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# Abstract

Powder technology (P/T) process includes compacting of fine powders followed by sintering process to get products of desired properties.

Several factors affected sintering; however, the most significant are time and temperature, with temperature being the most important single variable.

In this research study the effect of adding MgO on properties of SiO2- Al2O3 system . Many properties have been studied, such as .hardness, porosity, density and thermal conductivity was measured

Pure magnesia powder was mixed with different weight percent of alumina and silica. Four specimens were prepared, sample (1, 2, 3, 4) Sample (1) with (100%) alumina, sample (2) with ratio of (80%) alumina and (20%) silica, at last sample (3) with ratio of (40%) alumina, (20%) silica and (40%) magnesia, sample (4) with ratio of (40%) alumina and (60%) magnesia.

XRD tests and particle size were carried out for the powders.

Mixture of magnesia, alumina and silica were prepared by cold pressing at a pressure of (1) MPa, and sintering at temperature about (1200 °C) to obtain a very low porosity after final sintering, a good results have been got for the mechanical, physical and thermal properties.

To evaluate the performance of the prepared material, several tests were conducted such as hardness as a mechanical test. In addition, physical testes which include porosity and density, thermal properties such as thermal conductivity (thermal insulation) of these samples also were carried out.

The decrease in hardness of alumina  $(15.7 \text{ kg/mm}^2)$  compared to other samples (2,3,4) for about  $(15.5 \text{ kg/mm}^2, 15.4 \text{ kg/mm}^2, 15.6 \text{ kg/mm}^2)$ 

kg/mm<sup>2</sup>) respectively. Physical properties play good role in this study, porosity, density, were about  $(1\%, 2.9 \text{ g/cm}^3)$  respectively for sample (1), so the ratio of porosity about (0.80%, 0.89%, 0.96%) respectively for sample (2, 3, 4). Also this study shown the improvement in density that achieved for sample (1) by (2.9 g/cm<sup>3</sup>) and for sample (2, 3, 4) about (2.4 g/cm<sup>3</sup>, 2.6 g/cm<sup>3</sup>, 2.8 g/cm<sup>3</sup>) respectively.

Thermal conductivity for sample (3) is about (2.261 w/m.c) and for sample (4) is about (2.42 w/m.c), so the additives of Mgo to alumina cause lower thermal conductivity that is mean higher thermal insulation.

# **Chapter one** Introduction

# Introduction

# **1.1 Overview:**

Alumina (Al2O3), Silica (SiO2) and magnesia (MgO), are of great importance for applications in various machinery and transportation system, especially in thermal applications, aerospace, automobile products and several applications due to their superior physical and chemical performances.

# 1.2. Aluminium oxide

Aluminium oxide is a chemical compound of aluminium and oxygen with the chemical formula  $Al_2O_3$ . It is the most commonly occurring of several aluminium oxides, and specifically identified as aluminium(III) oxide. It is commonly called alumina and may also be called aloxide, aloxite, or alundum depending on particular forms or applications. It occurs naturally in its crystalline polymorphic phase  $\alpha$ -Al2O3 as the mineral corundum, varieties of which form the precious gemstones ruby and sapphire. Al2O3 is significant in its use to produce aluminium metal, as an abrasive owing to its hardness, and as a refractory material owing to its high melting point.

It is a naturally occurring common compound. It is useful in a variety of industries, most importantly in the manufacture of aluminium. The substance is used in the manufacture of industrial ceramics. Corundum, its most common crystal form, also has several variants of gem-quality.

# **1.3. Silicon dioxide**

It has been\_observed a very inhomogeneous dissociation of stoichiometric and non-stoichiometric thin SiO2SiO2 layers (thermally grown on SiSi substrates) during high temperature annealing at a low partial pressure of oxygen. During this process some silicon of the (100)Si(100)Si substrate and, in case of SiSi ion implantation, and additionally, excess SiSi is consumed. The SiO2SiO2 dissociation has been studied by electron microscopy and Rutherford backscattering spectrometry. Large holes (>1  $\mu$ m)(>1  $\mu$ m) in non-implanted oxide layers have been observed which evolve probably from defects located at the Si/SiO2Si/SiO2 interface. For SiSi implanted SiO2SiO2 additionally the formation of voids within the oxide during annealing has been observed preferably at the position of the implanted SiSi excess. Oxygen vacancies are possibly emitted from Si/SiO2Si/SiO2 interfaces into the oxide and migrate through SiO2SiO2 with long-range distortions of the oxide network. In that way the hole and void formation in the oxide can be explained by oxygen-vacancy formation, migration and siliconmonoxide (SiO)(SiO) emanation. As a driving force for growth of the large holes we identified oxygen diffusion from the Si/SiO2Si/SiO2 interface to the bare SiSi surface. This surface is a sink of oxygen diffusion due to the emanation of volatile.

## **1.4.** Magnesium oxide (MgO)

Magnesium oxide (MgO) and hydroxide  $[Mg(OH)_2]$  are conventionally considered insoluble in water and stable at high temperatures. However, in this study, we found significant dissociation of MgO and Mg(OH)<sub>2</sub> into ions when they were immersed in different physiologically relevant solutions in the form of 20-nm and 10-nm nanoparticles respectively, under standard cell culture conditions in vitro, i.e., a 37 °C, 5% CO<sub>2</sub>/95% sterile, humidified environment. The change in  $Mg^{2+}$  ion air. concentrations and pH measured in the physiologically relevant solutions (e.g., Dulbecco's modified Eagle's Medium (DMEM), simulated body fluid (SBF), relevant chloride solutions, and deionized water) confirmed their dissociation. Possible mechanisms and contributing factors for dissociation of MgO and Mg(OH)<sub>2</sub> nanoparticles were discussed. The evidence suggests that nucleophilic substitution of OH<sup>-</sup> by Cl<sup>-</sup> in Mg(OH)<sub>2</sub> is energetically unfavorable and it is more likely that Cl<sup>-</sup> plays a role in the stabilization of intermediate forms of MgO and Mg(OH)<sub>2</sub> as it dissociates. The pH and buffering capability of the immersion solutions might have played the most significant role in dissociation of these nanoparticles when compared with the roles of chloride (Cl<sup>-</sup>), proteins, and different buffering agents. This article provided the first evidence on the dissociation of MgO and Mg(OH)<sub>2</sub> nanoparticles in physiologically relevant conditions and elucidated possible factors contributing to the observed behaviors of these nanoparticles in vitro, which is important for their potential medical applications in vivo.

# **1.5. Objectives of the current study**

In view of facts mentioned above, the objectives of the current work are:

(1) Alumina was used as a basic material in this research, then proportions of magnesia and silica were added to know the effect of these materials on thermal conductivity.

(2) Study the effect of these additives on the several properties such as hardness, porosity and density.

(3) Study the thermal conductivity, which is a key property in this research, it is improved by supporting the other properties that were measured at the beginning.

### **1.6.** The aim

Aim and the main purpose of this research is to study the properties of the samples that were prepared in laboratories, ceramic as alumina was used as a basic material in this research, then amounts of magnesia and silica were added to know the effect of these materials on thermal conductivity.

then notice the development in mechanical, physical, and thermal properties.

The present research is an attempt to understand the influence of magnesia that adding to the mixture of alumina and silica.

# **Chapter two** Theoretical part

# **Theoretical part**

# **2.1.** Introduction

This chapter deals with the aim of study and main issues and the outline of characterization of mixing MgO nanosize with ratio of  $Al_2O_3 - SiO_2$  and study the properties of mixture.

# 2.2. Literature Revue

#### Magnesium oxide

(MgO), or magnesia, is a white hygroscopic solid mineral that occurs naturally as periclase and is a source of magnesium (see also oxide). It has an empirical formula of MgO and consists of a lattice of Mg<sup>2+</sup> ions and  $O^{2-}$  ions held together by ionic bonding. Magnesium hydroxide forms in the presence of water (MgO + H<sub>2</sub>O  $\rightarrow$  Mg(OH)<sub>2</sub>), but it can be reversed by heating it to remove moisture. Magnesium oxide (MgO) and magnesium hydroxide [Mg(OH)2] have been largely used in industrial applications (Amundsen et al. 2000; Fruhwirth et al. 1985; Johnson and Liu 2013), and the majority of literature on them focused on their microstructure, properties, and performance in non-biological environments. MgO is very stable at high temperatures in oxidizing and reducing atmospheres up to 2300 °C and 1700 °C, respectively and is a model for crystalline cubic structure, with a high degree of perfection (Amundsen et al. 2000). Its stability at these conditions led to the statements in most textbooks that MgO is generally "stable" and "relatively inert" as metal oxides (Atkins 2010; Cotton and Wilkinson 1988). However, the conditions under which MgO is "stable" and "relatively inert" were not clearly discussed. It is also well accepted that MgO is hygroscopic and hydrates at room temperature to form Mg(OH)2 (Láska et al. 1993; Mejias et al. 1999; Refson et al. 1995), which explains why MgO is rarely found in natural mineral deposits. The hygroscopic nature of MgO and its transition to Mg(OH)2 are often mentioned alongside a statement in textbooks that MgO is generally stable (Fruhwirth et al. 1985; Láska et al. 1993; Refson et al. 1995). Conventionally, Mg(OH)2 is known to be insoluble in water and stable at temperatures up to 300 °C (Amundsen et al. 2000). Recently, MgO and Mg(OH)2 have attracted increasing attention for biomedical applications because of their bioactivity (Fielding et al. 2014; Kum et al. 2014; Patel et al. 2013; Pourdanesh et al. 2014) and distinct effects on bone marrowderived mesenchymal stem cells (BMSCs) and pathogenic bacteria (Wetteland et al. 2016). MgO and Mg(OH)2 usually form at the interface of Mg-based biodegradable implants with cells and tissues as major degradation products, during passivation and degradation of metallic Mg and its alloys. Mg-based metallic alloys are a promising class of biodegradable metals for medical implant applications (Liu 2011; Lock et al. 2014; Tie et al. 2016). Clinically, MgO and Mg(OH)2 have long been used as a dietary supplements to improve bone density (Carpenter et al. 2006; Stendig-Lindberg et al. 1993). In physiological conditions, both MgO and Mg(OH)2 release magnesium ions (Mg2+), and Mg2+ ions demonstrated osteoconductivity in published works (Yoshizawa et al. 2014a; Yoshizawa et al. 2014b). MgO nanoparticles significantly increased BMSC adhesion and proliferation in low concentrations, e.g., at  $200 \,\mu\text{g/mL}$  (Wetteland et al. 2016) and exhibited antimicrobial properties (Sawai 2003; Sawai and Yoshikawa 2004; Wetteland et al. 2016), which is beneficial for reducing the risks associated with infections of implanted materials. However, the fundamental properties of MgO and Mg(OH)2 in physiologically relevant conditions, including their dissociation in the nanoparticle form, have not yet been well studied. It is necessary to build a library on the fundamental properties of MgO and Mg(OH)2 in physiological environments to further explore their applications in medicine. Specifically, the objective of this study was to investigate the dissociation of MgO and Mg(OH)2 nanoparticles when immersed in various physiologically relevant fluids under standard cell culture conditions in vitro, and characterize their changes in microstructure and composition. The standard in vitro cell culture condition, i.e., a 37 °C, 5% CO2/95% air, sterile, humidified environment was used in this study to elucidate why the behaviors of MgO and Mg(OH)2 nanoparticles in our previous in vitro cell culture experiments (Wetteland et al. 2016) differed from the solubility data found in standard chemistry literature. In this study, Dulbecco's modified Eagle's Medium (DMEM) was selected to represent widely used cell culture media. DMEM with or without supplement of serum proteins were included to determine whether the proteins could affect the dissociation of MgO and Mg(OH)2 in the media, which could provide important insight into the effects of proteins on the degradation process of Mg-based biodegradable metals because MgO and Mg(OH)2 represent the major intermediate degradation products of Mgbased biodegradable implants. Simulated body fluid (SBF) was selected to mimic human body fluid since the ion concentrations in SBF are equivalent to those found in human blood plasma (Oyane et al. 2003).

One of the buffers in SBF is 4-(2-hydroxyethyl)-1piperazineethanesulfonic acid (HEPES). HEPES buffer is a synthetic biological buffer that is effective in regulating pH between 6.8 and 8.2. HEPES regulated the local pH at the surface of Mg, which increased the degradation rate of Mg (Dezfuli et al. 2014; Ferguson et al. 1980). Considering the specific activity of HEPES, a solution of HEPES in water was included, to elucidate which component in SBF might have the greatest effect on MgO and Mg(OH)2 dissociation. It has been suggested in literature that Cl- is a key contributor to the corrosion of Mg-based metals by interacting with Mg(OH)2 at the surface of the metal. It was hypothesized that Cl- ions may initiate the dissociation of MgO and Mg(OH)2. To evaluate the effects of Cl- ions on the dissociation of MgO and Mg(OH)2, two solutions with different Cl- sources, MgCl2 and NaCl, were included in this study. The use of MgCl2 allowed for the delivery of Cl- without the addition of another component, considering that MgO and Mg(OH)2 are expected to release Mg2+ ions. NaCl should provide another excellent source of Cl- ions because previous studies showed that intermediate species of MgO formed in the solution with NaCl (Johnson and Liu 2013; Peng et al. 2014). Thus, NaCl was used as a comparison to MgCl2 to evaluate if the cations, Mg2+ versus Na+, would have different effects on dissociation of the nanoparticles. The concentration of the chloride salts was set at 103 mM, the same concentration of Cl- found in SBF. A higher concentration of MgCl2 at 200 mM was included to explore the possible effects of higher chloride concentrations on dissociation of MgO and Mg(OH)2. Deionized water (DI) was included as a control because it does not have physiologically relevant ions and proteins. Ion concentrations and ionic strengths for cDMEM, DMEM, SBF (Kokubo et al. 1990), HEPES, NaCl (103-mM Cl-), MgCl2 (103-mM Cl-), and MgCl2 (200-mM Cl-)

■Nano SiO2 and MgO particles were incorporated into  $\beta$ -tricalcium phosphate ( $\beta$ -TCP) scaffolds to improve the mechanical and biological properties. The porous cylindrical  $\beta$ -TCP scaffolds doped with 0.5 wt % SiO2, 1.0 wt % MgO, 0.5 wt % SiO2 + 1.0 wt % MgO were fabricated via selective laser sintering respectively and undoped  $\beta$ -TCP scaffold was also prepared as control. The phase composition and mechanical strength of the scaffolds were evaluated. X-ray diffraction analysis indicated that the phase transformation from  $\beta$ -TCP to  $\alpha$ -TCP was inhibited after the addition of MgO. The compressive strength of scaffold was improved from 3.12 ± 0.36 MPa ( $\beta$ -TCP) to 5.74 ± 0.62 MPa ( $\beta$ -TCP/SiO2), 9.02 ±

0.55 MPa ( $\beta$ -TCP/MgO) and 10.43  $\pm$  0.28 MPa ( $\beta$ -TCP/SiO2/MgO), respectively. The weight loss and apatite-forming ability of the scaffolds were evaluated by soaking them in simulated body fluid. The results demonstrated that both SiO2 and MgO dopings slowed down the degradation rate and improved the bioactivity of  $\beta$ -TCP scaffolds. In vitro cell culture studies indicated that SiO2 and MgO dopings facilitated cell attachment and proliferation. Combined addition of SiO2 and MgO were found optimal in enhancing both the mechanical and biological properties of  $\beta$ -TCP scaffold

■Metal oxides are widely used in adsorption technology as adsorbent surfaces because of their efficiency, low cost and unique physical properties. The aim of this review to clarify the role of aluminium oxide and Nano aluminium oxide in removing some chemicals contain that influence on human health such as dyes, antibiotics, and heavy metals. This paper also includes the affective of some adsorption parameters like pH, contact time, removal percentageand temperature. The Adsorption nature, kinetic adsorption models and isotherm models are also reported here.

The silico-ferrite of calcium and aluminum (SFCA) is a significant crystalline phase that bonds in high basicity sinter. Al2O3 and SiO2 play an important role in the formation of SFCA in the Fe2O3-CaO-SiO2-Al2O3 system, but the effect mechanism of Al2O3 and SiO2 on the formation of SFCA is unclear. To investigate this effect, sintering experiments were carried out with different temperatures and different times. It was found that the reaction of Al2O3 with CaFe2O4 (CF) as an initial product was easier to form during the calcium iron aluminum oxide (CFA) than that of SiO2 with CF to form SFC. This was due to the former directly forming to CFA while the latter initially formed Ca2SiO4 (C2S) and Ca2.5Fe15.5O25, and then SFC. It was also observed that when Al2O3 and SiO2 existed simultaneously, the Al2O3 initially reacted with CF to form CFA at 1100 °C, while the SiO2 participated in the formation of SFCA at 1150 °C without the formation of SFC. Moreover, it was understood that these were different effects in that the Al2O3 promoted the transformation from the orthorhombic crystal system to the triclinic crystal system, while the SiO2 dissolved into CFA to form the SFCA phase when Al2O3 existed. View Full-Text

# 2.3. properties of (Al2O3, SiO2, Mgo)

# 2.3.1. Properties of Aluminium Oxide (Al<sub>2</sub>O<sub>3</sub>)

#### 1. Reaction with sodium hydroxide

Aluminum oxide reacts with sodium hydroxide to produce sodium aluminate and water. This reaction takes place at a temperature of 900-1100°C. Salt and water is obtained in this reaction in which aluminium oxide acts as an acid.

 $A12O3 + 2NaOH \rightarrow 2NaAlO2 + H2O$ 

#### 2. Reaction with sulphuric acid

Metal oxides are generally basic in nature but aluminium oxide is amphoteric oxide.

Hence it acts both as acid and base. In this case it acts as a base

 $A12O3 + H2SO4 \rightarrow A12(SO4)3 + H2O$ 

This is a neutralisation reaction.

#### 3. Reaction with hydrochloric acid

Aluminum oxide contains oxide ions, and thus reacts with acids in the same way sodium or magnesium oxides do. Aluminum oxide reacts with hot dilute hydrochloric acid to give aluminum chloride solution.

 $Al2O3+6HCl \rightarrow 2AlCl3+3H2O$ 

# 2.3.2. properties of (Sio<sub>2</sub>)

transparent to gray, odorless, crystalline or amorphous solid. Its melting and boiling point are 1600 °C and 2230 °C, respectively. Its density is 2.65 g mL<sup>-1</sup>. It is insoluble in water and acid.Silicon dioxide nanoparticles appear in the form of a white powder. The table below provides the

Properties		
Chemical formula	SiO <sub>2</sub>	
Molar mass	60.08 g/mol	
Appearance	Transparent solid (Amorphous) White/Whitish Yellow (Powder/Sand)	
Density	2.648 (α-quartz), 2.196 (amorphous) g·cm <sup>-3[1]</sup>	
Melting point	1,713 °C (3,115 °F; 1,986 K) (amorphous) <sup>[1]:4.88</sup> to	
Boiling point	2,950 °C (5,340 °F; 3,220 K) <sup>[1]</sup>	
Magnetic susceptibility (χ)	–29.6·10 <sup>-6</sup> cm <sup>3</sup> /mol	
Thermal conductivity	12 (   c-axis), 6.8 (⊥ c-axis), 1.4 (am.) W/(m · K) <sup>[1]:12.213</sup>	
Refractive index (n <sub>D</sub> )	1.544 (o), 1.553 (e) <sup>[1]:</sup> 4.143	

physical properties of these nanoparticles

Silicon dioxide also known as silica, is an oxide of silicon with the chemical formula SiO<sub>2</sub>, most commonly found in nature as quartz and in various living organisms. In many parts of the world, silica is the major constituent of sand. Silica is one of the most complex and most abundant families of materials, existing as a compound of several minerals and as a synthetic product. Notable examples include fused quartz, fumed silica, silica gel, and aerogels. It is used in structural materials, microelectronics (as an electrical insulator), and as components in the food and pharmaceutical industries. When you look on a food or supplement label, chances are you'll see ingredients you've never heard of. Some you might not even be able to pronounce. Though several of these may make you feel hesitant or suspicious, others are safe, and it's merely their name that's off-putting.Silicon dioxide is one such ingredient. It's found in many products, though is often misunderstood.

Silicon dioxide (SiO<sub>2</sub>) is a natural compound made of two of the earth's most abundant materials: silicon (Si) and oxygen (O<sub>2</sub>).Silicon dioxide is most often recognized in the form of quartz. It's found naturally in water, plants, animals, and the earth. The earth's crust is 59 percent silica. It makes up more than 95 percent of known rocks on the planet. When you sit on a beach, it's silicon dioxide in the form of sand that gets between your toes. Silicon dioxide is most often recognized in the form of quartz. It's found naturally in water, plants, animals, and the earth. The earth's crust is 59 percent silica. It is 59 percent silica. It makes up more than 95 percent often recognized in the form of quartz. It's found naturally in water, plants, animals, and the earth. The earth's crust is 59 percent silica. It makes up more than 95 percent of known rocks on the planet. When you sit on a beach, it's silicon dioxide in the form of sand that gets between your toes. It's even found naturally in the tissues of the human body. Though it's unclear what role it plays, it's thought to be an essential nutrient our bodies need.

# 2.3.3. Properties of (Mgo)

- Atomic Symbol: Mg
- Atomic Number: 12
- Element Category: Alkaline metal
- Density: 1.738 g/cm<sup>3</sup> (20°C)
- Melting Point: 1202 °F (650 °C)
- Boiling Point: 1994 °F (1090 °C)
- Moh's Hardness: 2.5

Properties			
Chemical formula	MgO		
Molar mass	40.304 g/mol <sup>[1]</sup>		
Appearance	White powder		
Odor	Odorless		
Density	3.6 g/cm <sup>3[1]</sup>		
Melting point	2,852 °C (5,166 °F; 3,125 K) <sup>[1]</sup>		
Boiling point	3,600 °C (6,510 °F; 3,870 K) <sup>[1]</sup>		
Solubility	Soluble in acid, ammonia insoluble in alcohol		
Band gap	7.8 eV <sup>[2]</sup>		
Magnetic susceptibility ( <b>x</b> )	-10.2·10 <sup>-6</sup> cm <sup>3</sup> /mol <sup>[3]</sup>		
Thermal conductivity	45-60 W·m <sup>-1</sup> ·K <sup>-1[4]</sup>		
Refractive index $(n_{\rm D})$	1.7355		
Dipole moment	6.2 ± 0.6 D		

# 2.4. Applications

# 2.4.1. Applications of (Al<sub>2</sub>O<sub>3</sub>)

Known as alpha alumina in materials science communities or alundum (in fused form) or aloxite in the mining and ceramic communities aluminium oxide finds wide use. Annual world production of aluminium oxide in 2015 was approximately 115 million tonnes, over 90% of which is used in the manufacture of aluminium metal. The major uses of speciality aluminium oxides are in refractories, ceramics, polishing and abrasive applications. Large tonnages of aluminium hydroxide, from which alumina is derived, are used in the manufacture of zeolites, coating titania pigments, and as a fire retardant/smoke suppressant. Over 90% of the aluminium oxide, normally termed Smelter Grade Alumina (SGA), produced is consumed for the production of aluminium, usually by the Hall–Héroult process. The remainder, normally called speciality alumina is used in a wide variety of applications which reflect its inertness, temperature resistance and electrical resistance.

#### **Fillers**

Being fairly chemically inert and white, aluminium oxide is a favored filler for plastics. Aluminium oxide is a common ingredient in sunscreen and is sometimes also present in cosmetics such as blush, lipstick, and nail polish.

#### Glass

Many formulations of glass have aluminium oxide as an ingredient.[22] Aluminosilicate glass is a commonly used type of glass that often contains 5% to 10% alumina.

#### Catalysis

Aluminium oxide catalyses a variety of reactions that are useful industrially. In its largest scale application, aluminium oxide is the catalyst in the Claus process for converting hydrogen sulfide waste gases into elemental sulfur in refineries. It is also useful for dehydration of alcohols to alkenes. Aluminium oxide serves as a catalyst support for many industrial catalysts, such as those used in hydrodesulfurization and some Ziegler–Natta polymerizations.

#### **Gas purification**

Aluminium oxide is widely used to remove water from gas streams. Abrasive

Aluminium oxide is used for its hardness and strength. Its naturally occurring form, Corundum, is a 9 on the Mohs scale of mineral hardness (just below diamond). It is widely used as an abrasive, including as a much less expensive substitute for industrial diamond. Many types of sandpaper use aluminium oxide crystals. In addition, its low heat retention and low specific heat make it widely used in grinding particularly cutoff tools. operations, As the powdery abrasive mineral aloxite, it is a major component, along with silica, of the cue tip "chalk" used in billiards. Aluminium oxide powder is used in some CD/DVD polishing and scratch-repair kits. Its polishing qualities are also behind its use in toothpaste. It is also used in microdermabrasion, both in the machine process available through dermatologists and estheticians, and as a manual dermal abrasive used according to manufacturer directions.

## Aluminium oxide (Al<sub>2</sub>O<sub>3</sub> ) Uses

1. Aluminium oxide is one of the common ingredients in sunscreen and also present in cosmetics such as nail polish, blush, and lipstick

2. It is used in formulations of glass

3. It is used as a catalyst

4. It is used in the purification of water to remove water from the gas streams

5. It is used in sandpaper as an abrasive

6. Aluminium oxide is an electrical insulator used as a substrate for integrated circuits

7.Used in sodium vapor lamps

## **2.4.2.** Applications of Silicon Dioxide (SiO<sub>2</sub>) Nano Powder

Silica  $(SiO_2)$  nano powder is used to make flat glass, glass products, molten sand, cement, fiberglass, ceramic enamel, sandblasting for

antioxidants, filter sand, flux, refractory and light concrete. Silicon dioxide (SiO<sub>2</sub>) Nanoparticles is widely used in many industrial products. Rare crystals in nature can be used to create important parts of the electronics, optical instruments and crafts industry. Silicon dioxide (SiO<sub>2</sub>)nano powder is an important raw material for the manufacture of optical fibers. Generally, pure quartz can be used to make quartz glass. The coefficient of expansion of quartz glass is very small. It is equivalent to 1/18 of ordinary glass. It can withstand temperature change and acid resistance is good. Therefore, quartz glass is often used to make chemical instruments resistant to high temperatures. Quartz sand is often used as a glass material and as a building material.

The main applications and uses of Silicon dioxide (SiO<sub>2</sub>)Nanoparticles are stated below:

1. SiO<sub>2</sub> Nanoparticles as Adhesive and Sealer

Silica (SiO<sub>2</sub>)nano powder is the preferred material in the adhesive and sealant field. Adding it to the sealant can quickly form a net structure, inhibit colloidal liquid, increases speed, improves the effect of bonding, and because the particles are small, the sealing of the adhesive increases.

Moreover, due to the special development of hydrophobic silicon dioxide, structural adhesives belonging to advanced technologies have been produced. These structural adhesives allow the joining of different materials such as steel, aluminum, magnesium, and plastic.

Numerous processes can benefit from the technical advantages and improvements that can be made with the proper selection of dioxides that have been specially developed for the special adhesives and sealants industry. Silicon dioxide (SiO<sub>2</sub>)nano powder not only improves the mechanical and rheological properties but also acts as a counter to the solution of agents that improve the storage and processing stability of adhesives and sealants.

# 2.4.3.Applications of (Mgo):

#### Heating elements

MgO is prized as a refractory material, i.e. a solid that is physically and chemically stable at high temperatures. It has two useful attributes: high thermal conductivity and low electrical conductivity. Filling the spiral Calrod range top heating elements on kitchen electric stoves is a major use. "By far the largest consumer of magnesia worldwide is the refractory industry, which consumed about 56 % of the magnesia in the United States in 2004, the remaining 44 % being used in agricultural, chemical, construction, environmental, and other industrial applications. MgO is used as a basic refractory material for crucibles.

# Fireproofing

It is a principal fireproofing ingredient in construction materials. As a construction material, magnesium oxide wallboards have several attractive characteristics: fire resistance, termite resistance, moisture resistance, mold and mildew resistance, and strength.

#### Niche uses

MgO is one of the components in Portland cement in dry process plants. Magnesium oxide is used extensively in the soil and groundwater remediation, wastewater treatment, drinking water treatment, air emissions treatment, and waste treatment industries for its acid buffering capacity and related effectiveness in stabilizing dissolved heavy metal species.

Many heavy metals species, such as lead and cadmium are most soluble in water at acidic pH (below 6) as well as high pH (above 11). Solubility of metals affects bioavailability of the species and mobility soil and groundwater systems. Most metal species are toxic to humans at certain concentrations, therefore it is imperative to minimize metal bioavailability and mobility.

Granular MgO is often blended into metals-contaminated soil or waste material, which is also commonly of a low pH (acidic), in order to drive the pH into the 8–10 range where most metals are at their lowest solubilities (basic). Metal-hydroxide complexes have a tendency to precipitate out of aqueous solution in the pH range of 8–10. MgO is widely regarded as the most effective metals stabilization compound when compared to Portland cement, lime, kiln dust products, power generation waste products, and various proprietary products due to MgO's superior buffering capacity, cost effectiveness, and ease/safety of handling.

Most, if not all products that are marketed as metals stabilization technologies create very high pH conditions in aquifers whereas MgO creates an ideal aquifer condition with a pH of 8–10. Additionally, magnesium, an essential element to most biological systems, is provided to soil and groundwater microbial populations during MgO-assisted metals remediation as an added benefit.

### Medical

Magnesium oxide is used for relief of heartburn and dyspepsia, as an antacid, magnesium supplement, and as a short-term laxative. It is also used to improve symptoms of indigestion. Side effects of magnesium oxide may include nausea and cramping. In quantities sufficient to obtain a laxative effect, side effects of long-term use include enteroliths resulting in bowel obstruction.

#### Other

- As a food additive, it is used as an anticaking agent. It is known to the US Food and Drug Administration for cacao products; canned peas; and frozen dessert. It has an E number of E530.
- It was historically used as a reference white color in colorimetry, owing to its good diffusing and reflectivity properties. It may be smoked onto the surface of an opaque material to form an integrating sphere.
- It is used extensively as an electrical insulator in tubular construction heating elements. There are several mesh sizes available and most commonly used ones are 40 and 80 mesh per the American Foundry Society

# **2.5.** Compacting

Powder compaction can be summed up as the operation that provides shape, dimensional control, desired density and strength for subsequent handling. Conventional compaction occurs in the following manner, as shown in figure 2-1. Initially, as pressure is applied, the loose arrays of particles are packed closer and particle bridging is eliminated. At this stage the density attained is at most equivalent to the tap density. As the pressure is further increased the contact area grows through rearrangement and sliding. At high pressures the contact area increases through plastic deformation. The inter-particle bonds develop through cold welding, weak attractive forces and mechanical interlocking. The strength developed at this stage is called the green strength, while the pressed density is termed the green density. Generally, due to interlocking, rounded but irregular large shaped particles result in the highest green strength. The higher the hardness of the particles the lower the green density. If the particles are small, the finer pores will require higher pressures to collapse and the pressed density will be low. In general, characteristics that improve the green density is also improve the green strength .All methods of compaction have the same goal, which is to achieve the desired shapes with minimal die wall-tool friction. This resulted in reduced tool wear and improved pressing efficiency. Compaction to be successful (i.e. uniform compact properties), the ratio of compact height (H) to the compact diameter (D) must not exceed five .



# Figure 2-1 Stages of compaction, tap density, rearrangement and

#### green density.

The green-state compact consists of powder particles pressed together. Although the green-state compacts look to the eye like any solid part, there are no bonds in the compact other than interparticle bonds created through the deformational forces that pressed the powder particles together. Consequently, the green-state compact is very fragile and susceptible to damage.

Ceramic powders are commonly pressed into dies to produce nearnet shape "green" bodies prior to final sintering. Density gradients in the resulting compacts may cause distortion in the shape of the parts during sintering, necessitating expensive machining or grinding operations to obtain the desired final shape. Nonuniform shrinkage may even generate internal stresses that are sufficiently large to cause fracture of the part during sintering. Equally importantly, these density gradients can result in green bodies that break during ejection from the die or that are too fragile to be handled .

# **2.6. Sintering**

Sintering is a heating process in which a fine powder that has been formed into a shape is subsequently fired at temperature below melting point. The compact, when fired, densifies and becomes less or nonporous [10]. The powder particles form bonds at this temperature and the contact points between particles, which were originally formed under the mechanical pressure during compaction, increase in size and strength, improving mechanical properties.

The particle size has a great importance in P/T because it affects most of the properties. So the powders are divided into three distinct classes, sieve having the aperture of 44 micron, sub-sieve are smaller than the aperture of such a screen but grater than 1 micron and sub- micron or ultra-fine is smaller than 1 micron. Particle size is expressed by the diameter for spherical shaped particles and by the average diameter for non-spherical particles.

Sintering is a thermal treatment that densification accompanies. Densification almost always requires shrinkage (in some cases zero changes or even growth). The shrinkage takes place as a result of materials being transported by one or more of several diffusion processes. This may involve a liquid or reactive liquid or diffusion at grain boundaries or through the volume of particles .

Therefore, it is a densification process where porosity is usually reduced and the grain size increases significantly through mass transport. This is important because it allows an engineer to form a dense ceramic body at temperatures below the often very high melting temperature of the ceramic. For many properties such as strength, thermal conductivity and translucency, in order to reach a maximum value for a product. It is necessary to remove as much porosity as possible. A formed powder compact can be anywhere from 40% to 75% dense, with little strength. A sintered body can have much higher strengths and densities may exceeding 99%.

The ability to achieve dimensional tolerances in powder technology parts arises directly from the control of the sintering process and in particular shrinkage. Thus, a detailed understanding of the sintering process and the associated shrinkage is of considerable importance.

# 2.7. Sintering Mechanisms

Sintering proceeds from various mass-transport mechanisms. These can be divided into surface transport and bulk transport mechanisms. In surface transport mechanisms, atoms move from the surface of one particle to the surface of another particle. In bulk transport mechanisms, atoms move from the particle interior to the surface. Surface transport mechanisms lead to neck growth without shrinkage or densification, while bulk transport mechanisms result in net particle movement, leading to shrinkage and densification. Densification means an increase in packing density. The surface transport mechanisms are surface diffusion and vapor transport. While, the bulk transport mechanisms are lattice diffusion, grain boundary diffusion, and viscous flow. In powders composed of different materials, chemical reactions (also called reactive process) may also provide additional mass-transport mechanisms .

Different mechanisms dominate at different points in the sintering process and different materials exhibit different mechanisms. For instance, viscous flow is the diffusion in the liquid phase and evaporation condensation (vapor diffusion) is in the gaseous phase. Evaporation condensation and surface diffusion are called non-densifying mechanisms because they do not contribute to the pore shrinkage. They only increase the cohesion of the grains by the increase in the grain contacts. On the other hand, mechanisms contribute to the decrease of the volume fraction of pores; i.e.; the shrinkage .

The compaction powder (called a green compact) has a large surface area relative to its volume. This surface area provides the driving force in sintering, which is the reduction of free surface energy resulting from the high surface area of the particles .

Solid state sintering may performed by two alternative processes: namely one reduction of the total surface area by an increase in the average size of the particles, which leads to coarsening figure 2-3b, and two reduction by elimination of solid / vapor interface and the creation of grain boundary area followed by grain growth, which leads to densification figure 2-2a.



Figure 2-2 Schematic of two possible paths by which a collection of Particles can lower its energy .

These two mechanisms are usually in competition. If the ionic processes that lead to densification dominate, the pores get smaller and disappear with time and the compact shrinks. But if the atomic processes that lead to coarsening are faster, both the pores and grains coarsen and get larger with time .

Figure 2-3 shows neck growth with no shrinkage for simple array of spherical particles when the operating mechanism is a surface or vapor diffusion only. When a densifying mechanism is operating, both neck growth and densification take place as shown in the same figure.



Figure 2-3 a. Row of initially spherical particle b. Neck growth and no shrinkage by surface or vapor phase diffusion, and c. The shrinkage when a densifying mechanism is operating .

A sintered part begins as a green compact, which is low-density, inhomogeneous and porous, and generally lacks in physical integrity. There is, however, a small degree of adhesion between adjacent particles.

During the final stages of sintering, in addition to the elimination of pores, a general coarsening of the microstructure by grain growth may occur. During coarsening, smaller particles disappear and larger particles grow. The average grain size often related to the primary particle size. An exception to this is if there is grain growth due to long sintering times or exaggerated or abnormal grain growth. Typically, ceramics with a small grain size are stronger than coarse-grained ceramics.

Sintering can occur either at atmospheric pressure or under isostatic or hydrostatic pressure. This pressure-assisted sintering increases the sintering rate, reduces sintering time, and reduces porosity in the final part. Generally It takes place at temperatures in excess of half the absolute melting temperature. If sintering takes place at temperatures high enough to higher than melting point of one of the component of the elementary powders, it is called liquid-phase sintering, sintering that takes place at lower temperatures without any melting of any component is called solid-state sintering.

# 2.8. Sintering Stages

The kinetics of the process can be broken down into three sintering stages:

#### 2.8.1 Initial Neck Growth

Sintering initially causes the particles that are in contact to form grain boundaries at the point of contact through diffusion. This is the point contact stage and does not result in any dimensional changes. The greater the initial density of compaction (increased particle contact and potential grain boundary area), the higher the degree of coherency in the material. In this initial stage of sintering, necks begin to form at the contact points between adjacent particles figure 2-4. This stage is therefore referred to as the "neck growth" stage. No change in the dimensions is observed nor does porosity decrease .

Neck formation is driven by the energy gradient resulting from the different curvatures of the particles and the neck. Surface diffusion is usually the dominant mass-transport mechanism during the early stages of neck growth, as the compact is heated to the sintering temperature .



Figure 2-4 Neck formation .

#### 2.8.2 Intermediate Stage Sintering

Intermediate stage sintering begins when adjacent necks begin to impinge upon each other. This occurs when the quantity X/2R= 0.3, figure 2-4. Densification and grain growth occur during this stage. The packing density and coordination number of the green packing are important during this stage. A high green packing density produces rapid sintering with relatively few pores in the final object.

The intermediate stage is pore channel closure where interconnected pore channels are closed off isolating porosity. One of the causes of pore channel closure is neck growth. Another cause is the creation of new contact points by pore shrinkage within the pore itself.

Coarsening processes was detailed in figure 2-5, very low green packing densities (around 40%), which are also associated with low coordination numbers which was shown in figure 2-5-a and 2-5-b. This was leading to coarsening (increase in mean grain size) without densification (decrease in porosity). In extreme cases, this may lead to open-pore structures lacking in structural integrity, as shown in figure 2-6-c.



Figure 2-5 Coarsening resulting from low coordination number.

At the beginning of the intermediate stage, the pores form a network of interconnected cylindrical pores broken up by necks. By the end, the pores are smoother and begin to pinch off and become isolated from each other. Bulk transport mechanisms, such as grain boundary diffusion and volume diffusion, dominate the sintering process during this stage. As stated previously, these bulk transport mechanisms cause material to migrate from inside the particles to the surface, resulting in contact flattening and densification.

#### 2.8.3 Final Stage Sintering

Final stage sintering begins when most of the pores are closed. As sintering proceeds, the pores, which during intermediate stage sintering form a network, have become isolated from each other [14]. Final stage sintering is much slower than the initial and intermediate stages. As grain size increases, the pores tend to break away from the grain boundaries and become spherical. Pore shrinkage is the most important stage in sintering. For this stage to occur; solids must be transported into the pores and a means must exist by which the gas in the pores can escape to the surface. The resultant effect is to decrease the volume of the sintering mass. Grain boundary diffusion or lattice diffusion is usually the dominate mechanism during this stage .

Smaller pores are eliminated, while larger pores can grow, a phenomenon called ostwald ripening. In some cases, pore growth during final stage sintering can lead to a decrease in density, as gas pressure in the larger pores tends to inhibit further densification. This can be mitigated by having the final stage sintering occur into a partial vacuum.

## **2.9. Densification Rate Curves**

Densification rate curves as a function of relative density are sensitive to microstructure, such as initial parameters of microstructure (agglomeration, pore size, heterogeneities) and heating schedule (thermal pre-treatment, heating rate). Densification rate curves can be correlated with microstructural evolution during overall sintering and are expected to be a good help to choose raw materials. The development of the densification rate curve model, specifically its predictive ability, is an important step toward the realization of the concept of "materials by design", i.e. to create the desired materials through designs based on our understanding of materials.

Figure 2-6 illustrates the densification in three stages of sintering. The density-time curve in the Figure is composed of three distinct portions. The first portion reflects the initial stage where densification is due to only neck growth. The densification in the intermediate stag is due to concurrent grain growth and reduced number of interconnected pores. When the pores are completely in the closed form, the rate of densification is reduced as shown in the final stage .



Sintering Time →

Figure 2-6 Typical densification curve .

# **Chapter three**Experimental part

# **3.1. Introduction**

This chapter focuses on the preparation methods, materials, and equipment that were used in this study.

One of the most important production routes for ceramic or hard metal parts is powder technology by using pressing and sintering. Today, complex parts can be produced by (P/T) which were inconceivable just a few decades ago. Except for the very simplest part geometries, it is not possible to achieve a homogeneous green density distribution by die compaction. The density is more or less inhomogeneous depending on the part geometry, the tool design and the friction between powder and die wall.

After the mass of powder is squeezed into a shape and ejected from the press, it is fed slowly through a special high-temperature furnace to bond the particles together. They are metallurgically fused without complete melting, a phenomenon called "sintering"

# **3.2.Used Materials:**

The raw materials used in this study, as the main component is  $Al_2O_{3,}$ SiO<sub>2</sub> and MgO, with the ratio that occurs in table (1). Four samples were clarify in table below that examine in this research.

No. of sample	Al <sub>2</sub> O <sub>3</sub> %	SiO <sub>2</sub> %	MgO%
1	1 100		0
2	80	20	0
3	40	30	30
4	40	0	60

Table 1: ratio of the compenent

# **3.3:preparing Samples 3.3.1. preparing powder**

The experimental procedures to accomplish the objectives claimed in this research for producing mixture that shown in figure (3-1). The samples prepared with mixing the powder. The powder must be test by using the following measurement such as : XRD and Particle Size.



SiO<sub>2</sub> Figure (3-1) Powder of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, MgO

# 3.3.2. Mixing Powder

In this step mixing of the ttree powders was done by electric mixer for 4 hours to ensure good homogenous. The electric mixer contains of strong plastic vessel and knives rotate with different speeds as shown in fig.(3-2)



Figure (3-2) Electrical mixer

# 3.3.3. compacting

The mixture of powder was pressed into the form of a cylinder. Pressing serves not only to give the samples its final size and shape but performs several more important functions. The compression of the material necessarily decreases the proportion of voids .the pressing machine is available at the Babylon University / College of Materials Engineering – Department of Ceramics and Building Materials as shown in fig.(3-4) and the applied load that used in the pressing the samples was 1 Mpa.



Figure (3-3) Pressing method



Figure (3-4) Pressing machine

#### 3.3. 4. Drying

The next step after pressing was drying the samples in drying oven which is available at the Babylon University / College of Materials Engineering – Department of Ceramics and Building Materials at 110°C for 24 hours, as shown in fig.(3-5).



Figure (3-5) Drying oven

# 3.3.4. Sintering

The prepared green samples were sintered at (1220-1300) °C for soaking time of 1 hr. The rate of increasing temperature was chosen as 3°C/min. The cooling rate was chosen as 10°C/min.



#### Figure (3-6) Samples after sintering

# **3.4 Tests: 3.4.1: X-Ray Diffraction test**

X-Ray diffraction was used to characterize magnesia, alumina and silica powders by (XRD) type (Shimadzo, XRD6000, diffractometer, Japon: X-Ray are generated using copper (Cu-K) radiation at 30KV, 40 mA and wave length A-1.5406A°), radiation generating pattern of diffractions from powder sample at room temperature in 20 ranges of 20 to 80.

It is a rapid analytical technique, primarily used for phase identification of crystalline materials and also can provide information on unit cell dimensions. It is generated in cathode ray tube by heating a filament to produce accelerated electrons toward the target by applying high voltage. The interaction of the incident rays with sample produces constructive interference and diffracted ray when conditions satisfy the Bragg law

$$n\lambda = 2d\sin\theta \qquad (3-1)$$

Where  $\theta$ , is the angle of incidence of the x=ray.  $\lambda$ , is the wave length of the X-rays used and d is the spacing between atom layer. Figure (3-7 A) shows the apparatus of x-ray diffraction (XRD) type (Shimadzu 6000, Japan) which is available at the Babylon University / College of Materials Engineering – Department of Ceramics and Building Materials was used to characterize the structure of glass.

The measurement conditions of the x-ray diffraction were:

#### Measure Conditions X-ray Target : Cu Wave : 1.54060 (A) Voltage : 40.0 (KV) Current : 30.0 (mA)

#### Slit

Divergence : 1.0 (deg) Scatter : 1.0 (deg) Receiving : 0.3 (mm)

#### Measure

Axis : Theta- 2 theta Scan Mode : Continuous Scan Range : 20.0 – 59.0 (deg) Step : 0.02 (deg) Speed : 7.0 (deg/min ) Preset Time : 0.17 ( sec)

Subsequently, the powder of samples was prepared for XRD test aftermilling and sieving (mesh 200).



(A)

(B)

Fig (3-7) A-Shimadzu 6000 X-ray diffraction(XRD), B-Working principle.

#### 3.4.2. Particle Size

Battersize 2000 laser particle size analyzer as shown in Figure (3-8) was used to measure particle size of samples at the Babylon University / College of Materials Engineering – Department of Ceramic and Building Materials. The measuring range can be selected between (0.02µm- $2000\mu$ m). The principal operation of this device as shown in Figure(3-8) where during irradiation of the sample, a laser beam generates a diffraction pattern in which the laser light is deflected at various scatter angles depending on the grain size of the samples. Particles having the same size scatter at the same angle and the intensity of the scattered light provide information about the relative number of quantity of these particles. The angle and related intensity distribution are recorded by using a special multielement detector. A connected computer calculates the particles distribution in 31 channels according to grin size and percentage volume from diffraction patterns, which are generated during the measurement. According to these measurements, average particle size is determined. Fig (3-8) Battersize 2000 laser particle size analyzer.



# Fig (3-8) Battersize 2000 laser particle size analyzer

# 3.4.3: Hardness testing

Hardness test was conduct by using a vickers hardness method, Vickers devicetype (HVS-1000 China) as shown in figure (3-9). The load was used (100g) and time (15sec). The reading diameter rate was taken for each sample. The following law was adopted to calculate the Vickers hardness.

-----3-2 
$$HV = 1.8544 \times \frac{p}{D^2 average}$$

Where:

HV: Vickers hardness.

P: applied force (Kg/mm).

D: average diameter (um)



#### Figure (3-9) Vickers device

# 3.4.4. Porosity

Apparent porosity is the percentage ratio of the volume of the open pores to the bulk volume of the specimen. Whereas, Water absorption is the percentage ratio of the weight have absorbed water in the pores to the weight of the solid part of the specimen. The apparent porosity and water absorption was measured as Archimedes method.

$$P_{A} = [M-D]/[M-S] *100$$
 (3-3)

Where:

 $P_A = Apparent porosity.$ 

M = Saturated weight to the specimen (g) after 24h in water.

D = Dry weight of the specimen (g).

S = Suspended weight of the specimen (g).

$$Wa = [M - D]/D *100$$
 (3-4)

Where:

Wa = water absorption (%).



Figure(3-10) Boiling process

# 3.4.5. Density

Bulk density (B), in grams per cubic centimeter of a specimen is the quotient of its dry weight divided by the exterior volume; including pores :

B=D/V (3-5)

Where B is Bulk density, D is dry weight of the samples and V is the exterior volume of the samples.

# **3.4.6. Thermal Conductivity**

The thermal conductivity property (K) may vary locally with temperature, humidity, material composition, direction, etc. Knowledge of local thermal conductivity is important in the evaluation of heat transfer rates. A cylindrical mold is prepared to produce the required specimen with dimensions of (10 cm) diameter and (2cm) thickness for all samples.

Hot disk method is used for determining the thermal conductivity (K). The hot disk TPS 500 thermal constants analyzer shown in Fig. (3-11), quickly and accurately measures the thermal conductivity of a wide range of materials. The thermal conductivity test is conducted by Department of Materials Engineering Laboratories / University of Babylon.



Figure 3-11: The Hot Disk Device

# **Chapter FOUT** Result and Discussion

# **Result and Discussion**

# **4. Introduction**

This chapter discusses the results obtained for mixtures D and E selected in chapter three.

# 4.1. X-Ray Test

the X-R-D test worked for choose samples that contained mixing powder for increase thermal conductivity.

Figure(4.1) shown X-Ray diffraction of powder, Alumina , Silica and magnesia powder.





Figure (4-1) X-Ray diffraction of Magnesia, Alumina and Silica ppowder.

#### 4.2. Particle Size

The samples tested by using Bettersize2000 laser particle size analyzer, the results show that the particle size of magnesia is about 4.780  $\mu$ m and for alumina is about 3.977  $\mu$ m. Figure (4-2) shows the distribution of particles of Sample of MgO and figure (4-3) shows the distribution of particles of Sample of Al<sub>2</sub>O<sub>3</sub>.









#### **4.3.Hardness Test**

The results of Vickers hardness test are shown in (Table 4-1), and (Fig. 4-4) shows the relation between magnesia addition and Vickers's hardness. It can be observed that the hardness deceased by small degree significantly with the augmentation of the magnesia percentage.

No. of sample	Al <sub>2</sub> O <sub>3</sub> %	SiO <sub>2</sub> %	MgO%	Hardness Kg/mm <sup>2</sup>
1	100	0	0	15.7
2	80	20	0	15.5
3	40	30	30	15.4
4	40	0	60	15.6

Table (4-1) Results in Vickers's hardness





#### 4.4. Porosity Test

Table (4-2) shows the results of porosity for samples, and (Fig. 4-5) shows the relation between porosity with different addition of materials. The results show that the porosity decreased with decreasing ratio of alumina due to the different ratio of other materials which help in filling the pores of samples after sintering.

No. of sample	Al <sub>2</sub> O <sub>3</sub> %	SiO <sub>2</sub> %	MgO%	Porosity %
1	100	0	0	1
2	80	20	0	0.80
3	40	30	30	0.89
4	40	0	60	0.96

Table (4-2) results of porosity



Figure 4-5 : The relation between the samples and porosity

#### **4.5 Density Test**

The bulk density result of standard and mixing materials are shown in the table (4-3) and fig.(4-6), It can be noticed that density decreased by increasing the proportion of different addition. This behavior is expected due to the decline in porosity as mentioned above. the decrease in the density is the main reason behind the decrease in the mechanical properties such as hardness.

No. of sample	Al <sub>2</sub> O <sub>3</sub> %	SiO <sub>2</sub> %	MgO%	Density g/cm <sup>3</sup>
1	100	0	0	2.9
2	80	20	0	2.4
3	40	20	40	2.6
4	40	0	6	2.8

Table (4-3) Results of density





# 4.6. Thermal Conductivity

Thermal conductivity test was performed on prepared samples in order to determine the effect of magnesia with different ratio of alumina and silica by hot disk equipment.

Thermal conductivity test for samples that conducted by alumina 40% silica 20% and 40% magnesia in sample 3 and when adding ratios of magnesia powder have micro scale the thermal conductivity decreased.

The decrease in thermal conductivity means an increase in thermal insulation, and this is due to the fact that magnesia and alumina have high porosity in sample (4) compared to sample (3), which contains a percentage of silica that enters between the alumina and magnesia particles.

No. of sample	Al <sub>2</sub> O <sub>3</sub> %	SiO <sub>2</sub> %	MgO%	thermal conductivity w/m.c
3	40	30	30	2.261
4	40	0	60	2.42

Table (4-4) Results of thermal conductivity

# **Chapter Five** Conclusions and Recommendations

#### **5.1.**Conclusion

The following conclusions obtained are from the sintering process for mixing material.

1-Hardness values, of sample (1) is about (15.7 kg/mm) and for sample (2,3,4) were (15.5 kg/mm<sup>2</sup>, 15.4 kg/mm<sup>2</sup>, 15.6 kg/mm<sup>2</sup>) respectively which related directly to its density.

2- The value of porosity for composite materials is lower than pure material such as (1%, 0.80%, 0.89%, 0.96%) respectively for samples (1,2,3,4).

3- Improvement in density is achieved for sample (1) by  $(2.9 \text{ g/cm}^3)$  and for sample (2, 3, 4) were about  $(2.4 \text{ g/cm}^3, 2.6 \text{ g/cm}^3, 2.8 \text{ g/cm}^3)$  respectively.

4-Thermal conductivity values, for all samples were (2.261 w/m.c, 2.42 w/m.c) for samples (3,4) which related to porosity of material .

#### **5.2.Recommendations and Suggestions for Future Work**

Because of the importance of the composite material and for further work in this field for achieving better results, the following suggestions are presented.

- 1- Study the effect of different ratio of Al2O3-SiO2 with addition ratio of Zno on the physical chemical and mechanical properties .
- 2- Study the effect of Al2O3-SiO2 and MgO on the physical, chemical and mechanical properties by using vacuum sintering.

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