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Sintering behavior and thermal insulating properties of ($MgAl_20_4$-Al) spinel composite

A Thesis

Submitted to the College of Science, Al-Nahrain University in Partial Fulfillment of the Requirements for Degree of Master of Science in Physics.

By

Ali Hussein saleh

B.Sc., Al-Nahrain University (2014)

Supervised by

Dr. Qasid Abdual sattar Al-janabi

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بسم الله الرحمن الرحيم

قَالُوا سُبْحَانَكَ لَا عِلْمَ لَنَا إِلَّا مَا عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ ﴿٣٢﴾

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I certify that this thesis entitled ***"Sintering behavior and thermal insulating properties of Spinel-Al composite"*** was prepared by *"**Ali Hussein Saleh** "*, under our supervision at the College of Science - Al-Nahrain University as a partial fulfillment of the requirements for the Degree of Master of Science in Physics Science.

Signature:
Name: **Dr. Qasid Abdual sattar Al-janabi**
Scientific Degree: **Assistant Professor**
Date: **/ /2017**

In view of the available recommendation, I forward this project for debate by the examination committee.

Signature:
Name: **Dr. Saad Naji Abood**
Scientific Degree: **Professor**
Title: Head of Physics Science Department
College of Science / Al Nahrain University.
Date: **/ / 2017**

Committee Certification

We, the examining committee certify that we have read this thesis entitled "***Sintering behavior and thermal insulating properties of Spinel-Al composite*** and examined the student "***Ali Hussein Saleh***" in its contents and that in our opinion, it is accepted for the Degree of Master of Science in Physics Science.

Signature:
Name: **Dr. Aliyah Abdul Mhsin Shehab**
Scientific Degree: **Professor**
Date: /12/2017
(Chairman)

Signature:
Name: **Dr. Shihab Ahmed Zaidan**
Scientific Degree: **Assistant Professor**
Date***:*** **/12/2017**
(Member)

Signature:
Name: **Dr. Alaa Jabbar Gazai**
Scientific Degree: **Assistant Professor**
Date: **/12/2017**
(Member)

Signature:
Name: **Dr. Qasid Abdual sattar Al-janabi**
Scientific Degree: **Assistant Professor**
Date**: /12/ 2017**
(Member/ **Supervised**)

I, hereby certify upon the decision of the examining committee.

Signature:

Name: **Dr. Hadi M. A. Abood**

Scientific Degree: **Professor**
Title: **Dean of College of Science**
Date: **/ 12 / 2017**

Acknowledgment

Praise is to Allah lord of the creation. Best prayer and peace be unto, the prophet Muhammed messenger of Allah, and his pure holy descendant, and Nobel companions.

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Abstract

Spinel-Aluminum composites are drawing greater attention due to their low density, excellent physical, mechanical and refractory properties. This work aims to study the preparing one of the most current conventional technique and sintering behavior of Spinel-Al composite through characterization of the physical, mechanical properties as well as XRD and microstructure of the synthesis Spinel-Al composite specimens.

Magnesia (MgO) and alumina (Al_2O_3) were used as starting materials to prepare stoichiometric magnesium aluminate spinel (MA) via single stage firing process (solid state reaction).

Spinel (reinforced material) which prepared at sintering temperatures 1200^0C and 1400^0C for 2h soaking time was added in different weight percentage, i.e. 2,4,6,8 wt.% to the aluminum (matrix material) for synthesis Spinel-Al composites in a temperature range of $450\text{-}650^0$C via solid state reaction (SSR). The sintering parameters, XRD, microstructure, mechanical and thermal properties of the Spinel-Al composite products were investigated as well as the influence of each of the synthesized spinel contents and sintering temperature on sintering behavior of the composite products were also discussed. The results show that the better sintered density, water absorption, compressive strength, and thermal conductivity of the composite products obtained at low amount addition of spinel fired at 1200 and 1400^0C was 2wt.% and at 650^0C.

Also, studying of X-ray diffraction (XRD) for prepared spinel-aluminum composite specimens showed that the composites has many crystalline orientations (38.7^0, 44.97^0, 65.3^0 and 60.19^0), (39^0, 45.2^0, 65.62^0 and 60.33^0), (38.9^0, 45.15^0, 65.47^0, and 60.4^0) and (38.85^0, 45.26^0, 65.6^0 and 60.4^0) corresponding to (111), (200), (220) and (333) for 2,4,6,8wt% of spinel respectively.

The studying of the morphology of spinel-aluminum composite specimens reveal that average grain size of reaches about 6.03, 6.17, 6.22, 6.3 (µm) for 2, 4, 6, 8 wt.% of $MgAl_2O_4$ fired at 1200^0C respectively, whereas their average grain size reaches about 7.18, 7.57, 7.9, 8.48 (µm) for the same spinel content fired at 1400^0C respectively.

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List of Abbreviations

symbol	*Description*
CFRP	carbon fiber reinforced plastics
GFRP	glass fiber reinforced plastics
MMC	Metal-Matrix Composites
PM	powder metallurgy
CMCS	Ceramics Matrix Composites
PMCS	Polymer Matrix Composites
AMCs	Aluminum matrix composites
GB	grain boundary
CTE	Thermal expansion coefficient
SHS	self-propagating high temperature synthesis
MA	magnesium aluminate spinel
SEM	Scanning electron microscope
CCC	carbon- ceramic composites
XRD	X-ray diffraction

List of Symbols

symbol	*Description*	*Units*
σ	Compressive stress	N/mm^2
F	Load applied perpendicular to specimen crosses section	N
A^0	Cross sectional area (perpendicular to the force)	mm^2

ε	Compressive strain	Without
ΔL	Change in length	mm
L_0	The original length	mm
ρ_c	Density of composite material	g/cm^3
ρ_r	Density of reinforced material	g/cm^3
ρ_m	Density of matrix material	g/cm^3
V_r	Volume fraction of reinforced material	cm^3
V_m	Volume fraction of matrix material	cm^3
W.A	Water absorption	Without
W_s	Weight of sintered sample saturated in water	g.cm.s^{-2}
W_d	Weight of dry sintered sample	g.cm.s^{-2}

Chapter One

Introduction

Chapter one

Introduction

1.1 Introduction

Many of modern technologies require materials with combinations of properties that cannot be met by the conventional metal alloys, ceramics, and polymeric materials. By understanding of the dependence of the various type of composite behaviors on the characteristics, relative amounts, geometry distribution and properties of constituent phases, it is possible to design materials with better property combinations especially for those that are needed for aerospace, under water, and transportation applications.

Material property combinations have been extended by the development of composite material which is considered to be any multiphase material that exhibits a significant proportion of the properties of both constituent phases such that a better combination of properties is realized. Composites include multiphase metal alloy, ceramics, and polymers which are formed as a consequence of natural phenomena which is composed of just two phases; one is termed the matrix, which is continuous and surrounds the other phase, which is called the dispersed phase.

The properties of composites are a function of the properties of the constituent phases, their relative amount, and the geometry of the dispersed phase of particles and the particle size, distribution, and orientation [1].

As composites which produced from two or more materials or phase are used together to give a combination of properties that cannot be attained other stiffness, strength, weight, high-temperature performance, corrosion resistance, hardness, or conductivity, so, composites highlight focus how different materials can work in synergy. Abalone shell, wood, bone, and teeth are examples of naturally occurring composites. An example of a material that is a composite at the macro-scale is steel-reinforced concrete. Micro-scale composites include such materials as carbon or glass fiber reinforced plastics (CFRP or GFRP). These composites offer significant gains in specific strengths and are finding increasing usage in airplanes, electronic components, auto-motives, and sporting equipment [2].

In designing composite materials, scientists and engineers have ingeniously combined various metals, ceramics, and polymers to produce a new generation of materials which have been created to improve combinations of mechanical characteristics such as toughness, stiffness, and high temperature strength. There is an integrated effort in design, material, process, tooling, quality assurance, manufacturing, and even program management for composite to become competitive with metals. The composites industry has begun to recognize that the commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years [3].

Metal-Matrix Composites (MMC) is excellent candidate materials for advanced engineering systems. However, they have one major shortcoming that has limited their widespread use-their tendency to fracture easily. In many systems, the low ductility or brittleness of these composites is caused by micro failure processes that invariably begin at the interfaces. Thus, the mechanical behavior and the overall performance of metal-matrix composites are not limited by bulk properties or bulk phases, but by interface properties and toughness [4].

Metal-matrix composites (MMCs) include aluminum, magnesium, copper, nickel, and intermetallic compound alloys reinforced with ceramic and metal fibers. A variety of aerospace and automotive applications utilize MMCs. The metal matrix permits the composite difficult and expensive than producing the polymer-matrix materials [2].

1.2 Powder Metallurgy

The most common solid-state processing technique for synthesizing composite materials is powder metallurgy (PM) technique. Powder metallurgy is the process of blending fine powdered materials, compacting the same in to a desired shape or from inside a mould followed by heating of the compacted powder in a controlled atmosphere. Referred to as sintering to facilitate the formation of bonding of the powder particles to form the end product.

Fig. 1.1 shows four basic steps of P/M process [3].

1- powder manufacture

2- Mixing or blending of powders

3- Compacting of powders

4- Sintering

- As certain alloy combinations and cermet’s can only be made by PM, so raw materials for powder production can be pure elements or pure-alloyed powders.
- Properties of powder metallurgy products are highly dependent on the characteristics of starting powders.

Some of these important properties and characteristics are;

- Chemistry and purity
- Particle size and size distribution
- Particle shape
- Surface texture

- Powder metallurgy is the science of producing metal powders and making finished objects from mixed or alloyed powders with or without the addition of non-metallic constituent [5].

Raw material (metal or ceramic) in the form of powders is formed into specific shape and subsequently sintered at a temperature just below the melting temperature of the primary constituent (matrix material) of the powder mixture. PM technique has the capability of forming near-net-shaped products. Initial stage of this PM technique is blending where the powders (raw material) are mixed homogeneously. The blending conditions mostly depend on the density difference between the constituents of the powder mixture. After blending, cold press or hot press or hot isostatic press is employed to produce a “green compact,” which is about 80 % dense and then followed by sintering [6].

Powder metallurgy may be defined as the art of producing metal powders and using them to made useful objects. Powder metallurgy basics were used as far back as 3000 B.C. by the Egyptian to make iron implements. The use of gold, silver, copper, brass, and tin powders for ornamental purposes was common place during the middle ages [6].

Powder technologies are exciting to physist and engineers because processing options permit the selective placement of phases or pores to tailor the component for the application. The three main reasons for using PM are economic, uniqueness, and captive applications, as shown in Fig. 1.1 [1]. For some applications that require high volumes of parts with high precision, cost is the overarching factor. A good example of this segment is parts for the automotive industry (where approximately 70% of ferrous PM structural parts are used). Sometimes it is a unique microstructure or property that leads to the use of PM processing: for example, porous filters, self-lubricating bearings, dispersion strengthened alloys, functionally graded materials (e.g., titanium-hydroxyapatite), and cutting tools from tungsten carbide or diamond composites.

Captive applications of PM include materials that are difficult to process by other techniques, such as refractory metals and reactive metals. Other examples in this category are special compounds such as molybdenum disilicide and titanium aluminide, or amorphous metals.

The metal powder industry is a recognized metal forming technology that competes directly with other metalworking practices such as casting, forging, stamping (fine blanking) and screw machining [7].

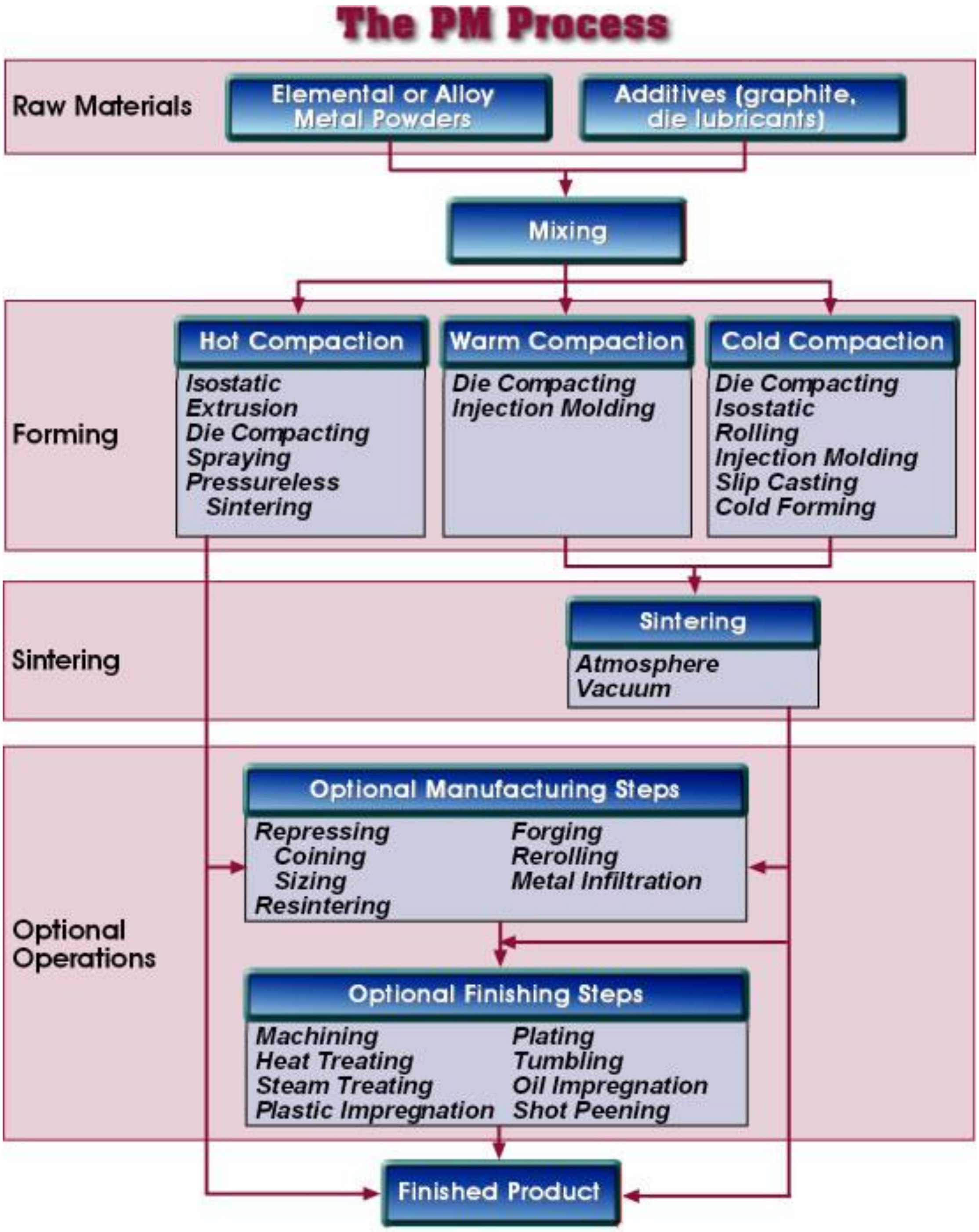


Figure 1.1 fabrication of composites by powder metallurgy techniques [8].

1.2.1 Metal powder

The performance of metal powders during processing and the properties of powder metallurgy products are highly dependent upon the characteristics of metals powder that are [9]:

- Purity
- Chemical composition
- Particle size
- Size distribution
- Particle shape
- Particle microstructure
- Apparent density
- Flow rate.

There are three main methods of powder production:

_ Mechanical, including machining, milling, and mechanical alloying.

_ Chemical, including electrolytic deposition, decomposition of a solid by a gas, thermal decomposition, precipitation from a liquid, precipitation from a gas, solid-solid reactive synthesis.

_ Physical, including atomization techniques.

The shape, size, size distribution, surface area, apparent density, flow, angle of repose, compressibility, and green strength depend on the powder production method [7].

Atomization is the method most frequently used for metals having low melting points, such as tin, lead, zinc, cadmium, and aluminum. As the liquid metal is forced through a small orifice, a stream of compressed air causes the metal to disintegrate and solidify into finally divided particles. Atomized products are generally in the form of sphere –shaped particles. A wide range of particle size distributions may be obtained by varying the temperature of the metal, pressure, and the temperature of the atomizing gas, rate of flow of the metal through the orifice, and the design of the orifice and nozzle. The principle advantage of the atomization process is its flexibility.

The reduction of compounds of the metal (usually an oxide) provides a convenient, economical, and flexible method of producing powders. The largest volume of metallurgical powder is made by the process of oxide reduction. Mill scale or chemically produced oxides are reduced with carbon monoxide or hydrogen, and the reduced powders are subsequently grounded. Oxide reduction is also an economical method of producing powders of iron, nickel, cobalt, and copper.

The method of electrolytic deposition is most suitable for the production of extremely pure powders of principally copper and iron. Powders obtained from hard, brittle electrodeposits are generally not suitable for molding purpose [6].

1.3 Composite materials

A typical composite material is a system of materials composing of two or more materials (mixed and bonded) on a macroscopic scale.

Generally, a composite material is composed of reinforcement (fibers, particles, flakes, and/or fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed properly, the new combined material exhibits better strength than would each individual material [10].

Jartiz [11], defined composites as a multifunctional material system that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form. While Berghezan [11] defines as “The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their shortcomings”, in order to obtain an improved material.

Van Suchetclan [12] explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

1.4 Characteristics of the composites

In all cases, the performance of the material during processing as well as the properties of the finished product depends to a large extent upon the basic characteristics of the powder material. Aside from the chemical composition and purity, the basic characteristics of a metal powder are particle size and size distribution, particle shape, apparent density, and particle microstructure.

Particle-size distribution is important in the packing of the powder and will influence its behavior during molding and sintering. For practical purposes, the selection of a desirable size distribution for a specific application is usually based upon experience. In general, a finer powder is preferred over a coarser powder, since finer powders have smaller pore size and larger contact areas, which usually results in better physical properties after sintering. Since the combination of dissimilar materials can have unique and very advantageous properties if the materials have appropriate characteristics, so the resulted material is better in certain key properties than either of the materials alone. The reinforcements and the matrix are usually very distinct types of materials with widely different properties [13].

Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the ‘reinforcement ‘or ‘reinforcing material’, whereas the continuous phase is termed as the ‘matrix’.

Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties which usually measured as volume or weight fraction to determine the contribution of a single constituent to the overall properties of the composites. It is not only the single most important parameter influencing the properties of the composites, but also an easily controllable manufacturing variable used to alter its properties [10].

1.5 Classification of composites

Composite materials can be classified in different ways:

- Classification based on the nature of matrix material into [8]:

1. Metal Matrix Composites MMCS
2. Ceramics Matrix Composites CMCS
3. Polymer Matrix Composites PMCS

- Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high performance of the composites. A typical classification is presented in Fig. 1.2. The two broad classes of composites are [3]:

- Particulate composites
- Fibrous composites

1.5.1 Particulate composites

As the name itself indicates, the reinforcement is of particle nature. It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a limited extent. Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage [3].

1.5.2 Fibrous composites

A fiber is characterized by its length being much greater compared to its cross sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might otherwise lead to failure [3].

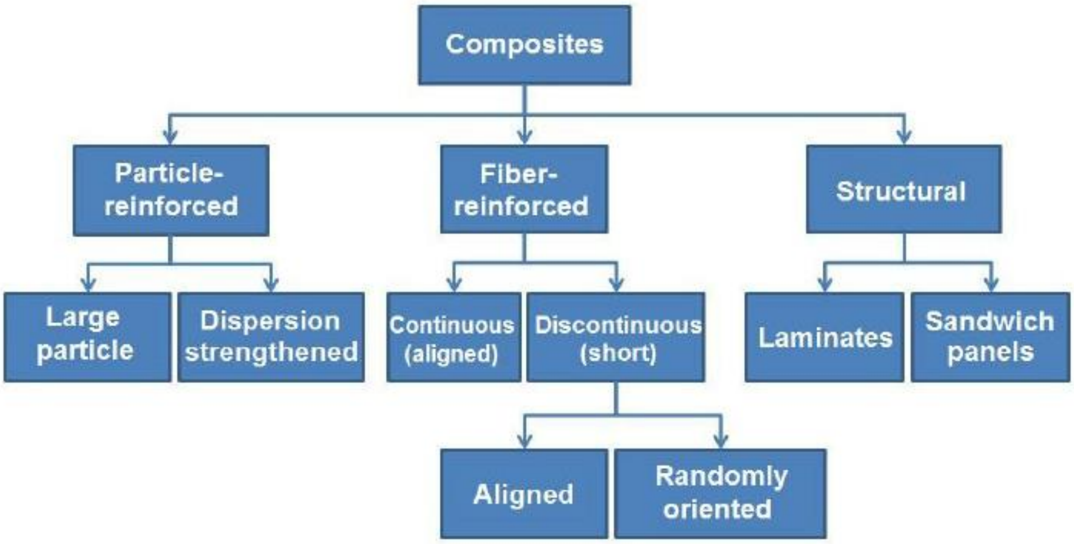


Fig. 1.2 classifications of the composites [3]

1.6 Metal–matrix composites (MMCs)

Metal matrix composites in general, consist of at least two components, one is the metal matrix and the second component is reinforcement. The matrix is defined as a metal in all cases, but a pure metal is rarely used as the matrix. It is generally an alloy. In the productivity of the composite the matrix and the reinforcement are mixed together.

In recent years, the development of metal matrix composite (MMCs) has been receiving worldwide attention on account of their superior strength and stiffness in addition to high wear resistance and creep resistance comparison to their corresponding wrought alloys. The ductile matrix permits the blunting of cracks and stress concentrations by plastic deformation and provides a material with improved fracture toughness [10].

The super alloys, as well as alloys of aluminum, magnesium, titanium, and copper, are employed as matrix materials. The reinforcement may be in the form of particulates, both continuous and discontinuous fibers, and whiskers [14].

Metal Matrix Composite (MMC) covers various types of system, and also a wide range of scales and microstructures. The reinforcing constituent is in most cases a ceramic. MMC types are commonly subdivided according to whether the reinforcement is in the form of [15]

- Particles
- Short fibers
- Long fibers.

Aluminum based alloys are widely used in applications where weight savings are important. However, the relatively poor wear resistance of Aluminum alloys has limited their use in certain high friction environments. The combination of properties and fabric ability of aluminum metal matrix composites makes them attractive candidates for many structural components requiring high-stiffness, high strength and low weight, good corrosion resistance, low density, and excellent mechanical properties [10].

MMCs are rising as a vital class of materials in the continuing search for improved strength, and stiffness in addition to other desirable properties, which make them competitive compared to monolithic metals. The specific properties obtained in MMCs will depend upon the exact matrix alloy and ceramic selection, the form, size and percentage of the ceramic added, ceramic properties, interface properties and the processing method selected to produce the composite. MMC is normally fabricated using a ductile metal (e.g. Aluminum, titanium and nickel) as the matrix, ceramic as the reinforcement (e.g. alumina, Silicon carbide, and graphite), which combines the good ductility and toughness of the metal matrix with the high strength, hardness, and elastic modulus of the ceramic reinforcement.

Most of the related MMC research is aluminum based MMC with ceramic as the reinforcement. So aluminum alloys are chosen as a matrix material because of their low density, wide alloy range, heat treatment capability and processing flexibility. Moreover, Al based MMCs offers advantage of lower cost over most other MMCs and has excellent thermal conductivity, high shear strength, and ability to be formed, compared to the unreinforced Al alloys. The ceramic reinforcement materials, such as SiC, B_4C, nitrides, and Al_20_3 are in the form of continuous fibers, whiskers, platelets, or particles [16].

1.7 Literatures review

Aluminum matrix composites (AMCs) reinforced with spinel ceramics are of interest in many industrial applications due to the combination of excellent properties of Al matrix and the reinforced particulate, such as high specific strength and stiffness, good water resistance, elevated temperature stability, and low cost of production [17-18].

Various ceramics and intermetallic have been used as reinforcing materials to improve aluminum matrix properties such as Ni_3Al [19], TiB_2[20], $Al_{18}B_4O_{33}$[21] and Sic [22,23]. Among these compounds, the $MgAl_2O_4$ spinel is a novel reinforcement for AMCs due to a series of unique properties such as high young's modulus and strength, low thermal expansion coefficient (CTE) [24,25]. The use of synthesized $MgAl_2O_4$ spinel by a single-firing stage method without extra additives to reinforce Al matrix could reduce the cost of Al matrix composite.

According to previous studies, the addition of reinforcements such as ceramic particles, can apparently improve the overall properties of the composites, many of the conventional fabrication method of pure Aluminum matrix composites (AMCs) causes some problems, such as poor distribution of reinforcements in matrix, weak interface bonding between matrix and reinforcements, etc. Thus, new fabrication methods for composites need to be developed to improve their properties. AMCs fabrication achieved by adding synthesized reinforcements to the matrix and observing the wettability between the matrix and reinforcement, homogeneous distribution of reinforcement and lower cost of production compared with the other fabrication techniques.

$MgAl_2O_4$ spinel reinforcement is a kind of novel ceramic material with good mechanical strength, thermal shock resistance, excellent chemical inertness, and has found its application in metallurgical, electronic and chemical industries [26].

Giovane Azevedo et al, in 2000 [27] studied the Synthesis and characterization of Aluminum–Zirconium Intermetallic Composites. They conclude that The effect of increased control over the processing parameters has had a beneficial impact on both the integrity of the composite and the mechanical properties.

D. Horvitz, I. Gotman, in 2002 [28] Studied self-propagating high temperature synthesis (SHS) of $MgAl_2O_4$ – TiAl composite. They observed wave-like propagation of both leading and lagging reactions during thermal explosion.

S. Asavavisithchai et al, in 2006 [29] studied the effect of Mg addition on the stability of Al–Al_2O_3 foams made by a powder metallurgy route and they notice that the wetting is affected by reaction between Mg and alumina at the metal–oxide interface, forming $MgAl_2O_4$ which is thought to modify and rupture the oxide film on the surface of the molten Al exposing clean, wettable surfaces.

Kerti and Toptan, in 2007 [30] investigated microstructural variations in cast B_4C reinforced Aluminum Matrix Composites (AMCs). They concluded that B_4C addition with bigger particle size resulted in better microstructure with free of agglomerated particles.

Abenojar et al, in 2007 [31] investigated optimization of processing parameters for the Al + 10% B_4C system obtained by mechanical alloying. They found that the best results of hardness at different temperatures were obtained at 635ºC and argon atmosphere as sintering conditions.

Mu-yun lei et al, in 2007 [32] investigated the effect of HIP on the properties and microstructure of transparent polycrystalline spinel. They found after HIPing, the strength of the sample decreased, the green size increased, and the transmittance and the uniformity of transmittance increased significantly.

H.S. Tripathi et al, in 2009 [33] studied the Synthesis and densification behavior of magnesium aluminate spinel: Effect of Dy_2O_3 and they found that Dy_2O_3 additive does not have significant effect on the spinel isation but favors the densification of the spinel.

Salvador et al, in 2009 [34] studied high temperature mechanical properties of aluminum alloys reinforced with boron carbide particles. Tensile properties and fracture analysis of these materials were investigated at room temperature and at a high temperature. They observed that the mechanical properties have been improved by adding B_4C particles as reinforcement, and also the tensile strength of Aluminum Matrix Composites (AMCs) with B_4C decreases as the temperature increased.

Ahmed A. Moosa, in 2010 [35] studied the Microstructure and Some Properties of Aluminum-Silicon Matrix Composites Reinforced by Alumina or Chromia The squeeze casting technique produces parts with reduced porosity levels because of the high pressure applied during solidification and due to efficient liquid feeding obtained by moving the ram to compensate for the freezing contraction.

J. Wojewoda-Budka et al, in 2010 [36] studied the Interactions between molten aluminum and Y_2O_3 studied with TEM techniques. Their conclusion was The interaction between aluminum and yttria at 1273 K led to significant alloying of Al droplet with Y up to hypereutectic composition and to the formation of multilayer reaction product region within the Y_2O_3 substrate.

N.M.Khalil et al, in 2010 [37] studied the improve the sintering, mechanical and refractory properties of magnesium aluminate spinel, magnesia–spinel and alumina–spinel refractory bodies through addition of chromia, zirconia or their mixtures. . Magnesium nitrate and aluminum nitrate were used to prepare magnesium aluminate spinel (MA) via a co-precipitation technique while aluminum hydroxide and magnesium chloride were used to prepare magnesium aluminate spinel (MA) via sol–gel technique. Were the preparation of high purity magnesium aluminate spinel using a low cost-effective route is important. The solid state synthesis of MA, as a conventional synthesis technique, is used. In this technique, the reaction in the solid phase takes place between high purity MgO and Al_2O_3 over a temperature range of 1200–1400°C for commercial practice.

Chao Sun et al, in 2011 [38] investigated the effects of particle size and particle distribution on the microstructures and mechanical properties of Sic-reinforced pure Al composites have been studied before and after extrusion. Scanning electron microscope (SEM) and mechanical testing were used to characterize the microstructures and mechanical properties of the composites.

Natalya Froumin et al, in 2012 [39] studied the interfacial interaction between quasi-binary oxides ($MgAl_2O_4$ and $Y_3Al_5O_{12}$) and liquid aluminum by the sessile drop method in conjunction with a thermodynamic analysis in order to account for the experimental results.

Yang Zhou et al, in 2013 [40] studied the microstructure and properties of $MgAl_2O_4$ spinel whisker reinforced composite by a powder metallurgy technique. Their measurements show that $MgAl_2O_4$ whisker reinforced Al composites exhibit better thermal stability and lower thermal expansion coefficient than aluminum alloys.

L. M. Manocha et al, in 2013 [41] developed carbon- ceramic composites (CCC) for friction applications by using different types of reinforcements in the form of particulate and fibers with phenolic resin as carbon matrix precursor.

K. Moritz et al, in 2014 [42] studied the thermal shock resistance of alumina-rich magnesium aluminate spinel refractory ceramics with aluminum titanate as second phase. They found that aluminum titanate was higher than that of the pure spinel samples. And their thermal shock resistance was improved.

Yang Zhou et al, in 2014 [43] study processing and behaviors of $MgAl_2O_4$ spinel whiskers reinforced 6061 Al composite. The result show that the whiskers exhibit a homogeneous distribution with good interface bonding with the matrix. The hardness and tensile strength increase with increase content of whisker.

Tateoki IIZUKA et al, in 2014 [44] investigated the effect of the average size and the size distribution of $MgAl_2O_4$ particles on the dispersibility of AC_4C aluminum composites which were fabricated by the stirring−casting method. Microstructures, strength, and fatigue properties of $MgAl_2O_4$ particle-reinforced AC_4C based alloy composites were evaluated.

Juntong Huang et al, in 2014 [45] studied the preparation, microstructure and mechanical properties of spinel-corundum-sialon composite materials, this work could provide a new method to reduce environmental pollution and manufacture low cost high performance refractory materials using the abundant waste materials fly ash and aluminum dross.

G. Cevikbas et al, in 2015 [46] studied the properties of Aluminum titanate (Al_2TiO_5)-Spinel ($MgAl_2O_4$) ceramics composites. Its behavior in Radiation show that the Al_2TiO_5-$MgAl_2O_4$ ceramics composites can be used for nuclear shielding applications.

1.8 Aim of Research

The present work focuses on the ability of preparation of $MgAl_2O_4$-Al composite via one of the conventional method (solid state reaction by single stage firing process) and investigated its properties. The aim of this thesis is to study the sintering behavior of this kind of ceramic-metal composite. The main objectives of this project are shown below:

1- Study the effect of the percentage weight of Spinel on the physical, mechanical, thermal, and microstructure of Spinel-Al composite.

2- Specify the best percentage weight of Spinel content which can be added to obtain best properties of Spinel-Al composite.

3- Study the effect of sintering temperature of synthesized spinel on the properties of Spinel-Al composite.

4- Enhance the thermal conductivity of Aluminum upon addition of sintered Spinel.

Chapter Two

Theoretical part

Chapter Two

Theoretical part

2.1 Introduction

Pure metal matrix such as pure Aluminum is being used as candidate matrix element for the composites fabrication in areas where weight-saving and high strength are of major concern [47]. The addition of reinforcements such as ceramic particles can improve the overall properties of the composites [48,49]. Fabrication methods of pure Aluminum matrix composites (AMCs) are the ex-situ process, that is, Spinel reinforcement are added in to the Aluminum matrix and mixed together, then compacted and sintering according to powder metallurgy technique [50-52].

2.2 Powder compaction

The principle goal of the compaction process is to apply pressurize and bond the particles to form a cohesion among the powder particles. This is termed as the green compact. The compaction process imparts the following effects.

1. Reduces voids between the powder particles and enhance the density of the consolidated powder.
2. Produces adhesion and bonding of the powder particles to improve green strength in the consolidated powder particles.
3. Facilitates plastic deformation of the powder particles to conform to the final desired shape of the compact.
4. Enhances the contact area among the powder particles and facilitates the subsequent sintering process.

Compaction is carried out by filling a measured amount of metallic powder the die cavity and applying pressure by means of one or more plungers. To improve uniformity of pressure and reduce porosity in the compacted part, compressive forces from both the top and the bottom sides are necessary. The requisite compacting pressure depends on the specific characteristics and initial shape of the particles, the method of blending and the application of the lubricants. Extremely hard powders are slower and more difficult to compact. Some organic binder is usually required to hold the hard particles together after compacting until the sintering process is performed. Figure 2.1 depicts a schematic view of the powder compaction process to manufacture a typical product [5].

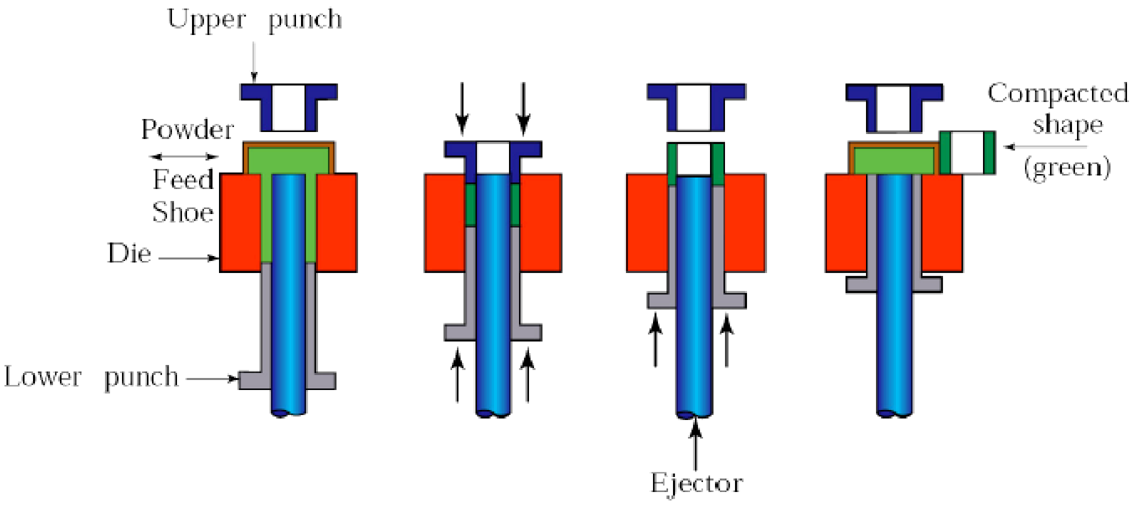


Figure 2.1 Compaction of metal powder to form a product [5]

Compacting techniques may be classified into two types [9].

- Pressure techniques such as die, isostatic, high energy-rate forming, forging extrusion, vibrating, and the continuous
- Pressure less techniques such as slip casting, gravity, and the continuous.

2.3 Sintering Theory

In 1922 Sauerwald was proposed the first theory of sintering. This theory says that two stages are involved in sintering namely adhesion and recrystallization. Adhesion occurs during heating due to atomic attraction whereas recrystallization occurs at recrystallization temperature (above 50 T_m). In recrystallization, microstructure changes, phase changes, grain growth, shrinkage occurs.

Sintering may involve [53]:

1- Single component system; self-diffusion is the major material transport mechanism and the driving force resulting from a chemical potential gradient due to surface tension and capillary forces between particles.

2- Multi-component system (more than one phase); inter-diffusion occurs with the concentration gradient being the major driving force for sintering in addition to self-diffusion caused by surface tension and capillary forces. In this sintering, liquid phase formation and solid solution formation also occurs with densification.

2.4 Sintering process:

Sintering is a process of consolidating either powder or a green compact of the desired composition under controlled conditions of temperature and time [54].

There are four types of sintering

1- Solid state sintering

2- Liquid phase sintering

3- Activated sintering

4- Reaction sintering

1- **solid state sintering**: in this process, densification occurs mainly because of atomic diffusion in solid state

2- **liquid phase sintering**: in this process densification is improved by employing a small amount of liquid phase (1-10 vol.%). The liquid phase existing within the powder at the sintering temperature has some solubility for the solid-during sintering, there is a rapid rearrangement of solid particles leading to density increase. So, solid phase sintering occurs resulting in grain coarsening and densification rates slows down.

3- **Activated sintering**: in this process, small amount of doping (alloying element) is added to improve the densification by as much as 100 times than undoped compact samples.

4- **Reaction sintering**: in this process, high temperature materials resulting from chemical reaction between the individual constituents, giving very good bonding. So, reaction sintering occurs when two or more components react chemically during sintering to create end product.

2.4.1 Solid state sintering

During sintering, densification occurs and solid state sintering is carried out at temperature where material transport due to diffusion is appreciable. This occurs by replacing high energy solid-vapour interfaces (with free energy γ_{SV}) with the low energy solid-solid interface (particle-particle) of free energy γ_{SS}. This reduction in surface energy causes densification [55].

Initially; $\gamma_{SS} < \gamma_{SV}$

The process of sintering will stop if (dE) becomes zero;

i.e. $dE = \gamma_{SS}\, dA_{SS} + \gamma_{SV}\, dA_{SV} \leq 0$ -----------------(2.1)

Where dE= over all changes in free energy of the system

dA_{SS}= interfacial area of solid-solid interface

dA_{SV}= interfacial area of solid-vapour interface

Initially, the surface area of compact represents the free surface area, since no grain boundaries have developed and hence $A_{SV} = A_{SV0}$ & $A_{SS} = 0$.

As sintering proceeds, A_{ss} increases and A_{SV} decreases. The sintering process will stop when dE = 0,

i.e. $\gamma_{SS}\, dA_{SS} + \gamma_{SV}\, dA_{SV} = 0$ ----------- (2.2)

$\gamma_{SS} / \gamma_{SV} = -\, dA_{SV} / dA_{SS}$ -------------(2.3)

Densification stops when $-\, dA_{SV} / dA_{SS}$ is close to zero

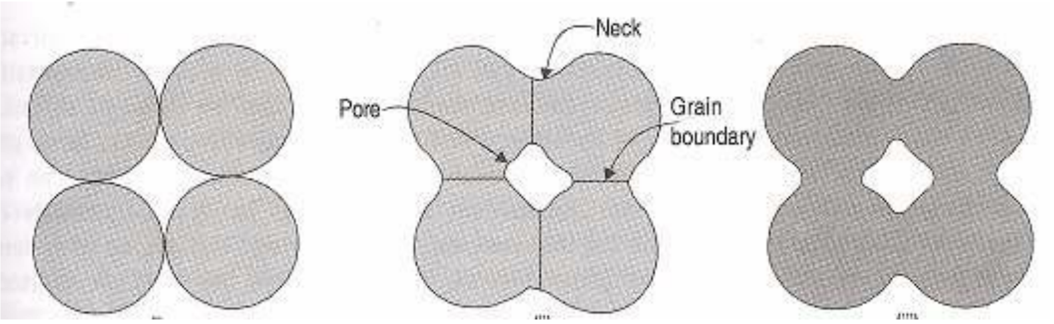
2.4.2 Solid state sintering stages

Three main stages are observed in solid state sintering [56]:

1- **First stage**: Necks are formed at the contact points between the particles, which continue to grow. During this stage, pores are interconnected and the pore shapes are irregular.

2- **Second stage**: with neck growth, the pore channels become more cylindrical in nature. With time at the sintering temperature, the pore eventually becomes rounded. With continued sintering, a network of pores and a skeleton of solid particle is formed.

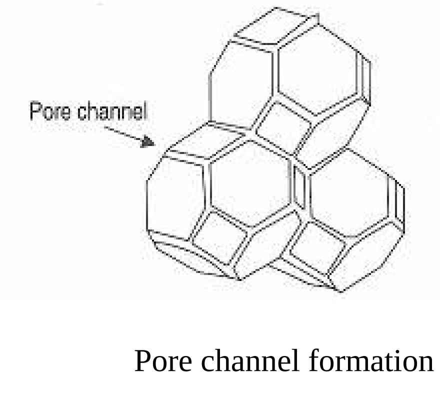
3- **Third final stages**: in this stage, pore channel closure occurs and the pores become isolated and no longer interconnected.



Particles in contact

Formation of necks, grain boundaries, pores

Final sintered geometry



Pore channel formation

Fig. 2.2 Shows solid-state sintering stage [56]

2.4.3 Liquid phase sintering

Sintering of multi-component systems may involve liquid phase formation, if the powder aggregate consists of a low component whose melting point is below the sintering temperature, where the formed liquid phase during sintering aids in densification of the compact. As liquid phase sintering employs a small amount of a second constituent having relatively low melting point, so it helps to bind the solid particles together and also aids in densification of the component. Such process is widely used for ceramics and refractories [57].

2.4.3.1 Liquid state sintering stages

1. Initial solid particles rearrangement occur once the liquid phase is formed. These particles flow under the influence of surface tension forces [5].

2. Solution & reprecipitation process: in this stage, smaller particles dissolve from areas where they are in contact which causes the particle centers to come closer causing densification. Whereas, the dissolved material is carried away from the contact area and reprecipitate on larger particles.

3. Solid state sintering.

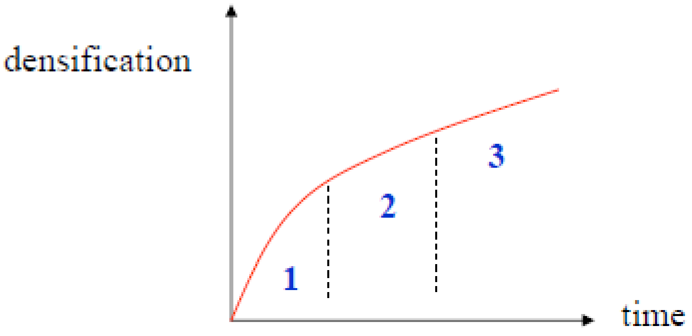


Fig. 2.3 The three stages densification of liquid phase sintering [5]

2.5 Property change during sintering

In the case of single component system, densification is proportional to the shrinkage or the amount of pores removed, whereas, in multicomponent system, expansion rather than shrinkage will result in densification and hence densification cannot be treated as equal to the amount of porosity removed.

Therefore, densification result in mechanical property change (hardness, strength, and toughness), physical properties (electrical, thermal conductivity, and magnetic properties) [5].

Also change in composition is expressed due to the formation of solid solution.

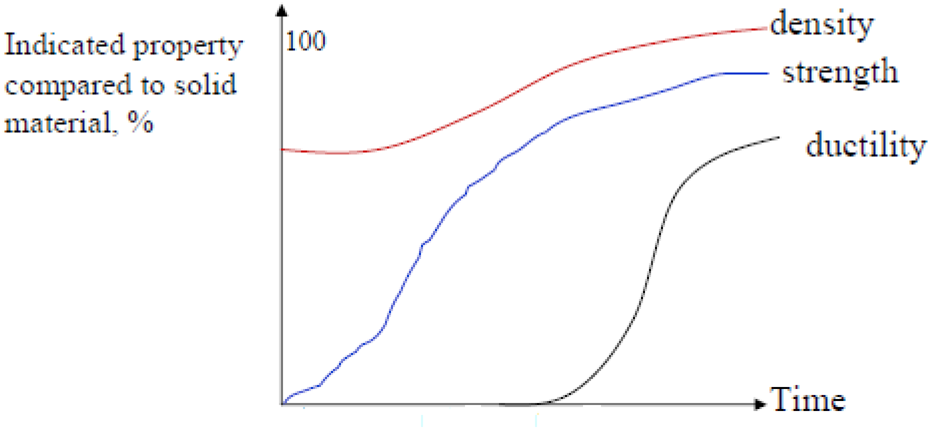


Fig. 2.4 Effect of change in composition during densification resulting in some mechanical property change [5]

2.6 Sintering process of metal powder

Heat treatment is an operation or combination of operations involving heating and cooling of a metal/alloy in solid state to obtain desirable [9];

- Properties, e.g., better machinability, improved ductility, and homogenous structure.
- Conditions e.g., that of relived stresses

The purpose of heat treating of aluminum alloys is to:

- Raise their strength and hardness
- Improve ductility
- Improve mechanical properties of both cast and wrought alloys.

Sintering consist of heating pressed metals or cermet compacts in batch or continuous furnaces to a temperature below the melting point of the major constituent in an inert or reducing atmosphere (of hydrogen, dissociated ammonia or cracked hydrocarbon), where time, temperature, heating rate and cooling rate are automatically controlled.

Sintering is done to achieve all possible final strength and hardness needed in finished product.

Most metals are sintered at 70 to 80% of the melting temperature. The sintering time varies from thirty minutes up to several hours'. Sintering temperatures and time varies considerably with different materials. Sintering is essentially a process of bonding solid bodies (particles) by atomic forces [58].

The process of sintering is generally the result of atomic motion stimulated by the high temperatures. The initial strains, surface area, and curvatures in a pressed powder compacts drive the atomic motions responsible for sintering. Several different patterns of atomic motion can contribute to the effect including evaporation and condensation, volume diffusion, grain boundary and surface diffusion, and plastic flow. In most cases the sintering kinetics are determined by several parameters including pressed density, material, particle size, sintering atmosphere, temperature and even the degree of sintering. As material transported takes place, the geometric progression can be divided into a number of stages representative of driving forces [48]:

- Initial bonding among particles
- Neck growth
- Pore channel closure
- Pore rounding
- Pore shrinkage
- Pore coarsening

Fig. 2.5 shows how pore structure changes with sintering. In the latter stage of sintering, interaction between pores and grain boundaries can take one of the following forms: the pores can retard grain growth, then can be dragged by the moving grain boundaries during grain growth or the grain boundaries can break away from the pores, leaving them isolated in the grain interior. Separation of the pores from the boundaries limits the final sintered density. It's therefore important to minimize breakaway by careful temperature control during sintering. The selection of a correct isothermal sintering temperature is important in successful densification. Higher temperatures lead to faster densification, but the rate of coarsening also increases. This increased coarsening rate may lead to abnormal grain growth with pores trapped inside large grains. Thus although densification proceeds faster, the final density may be limited.

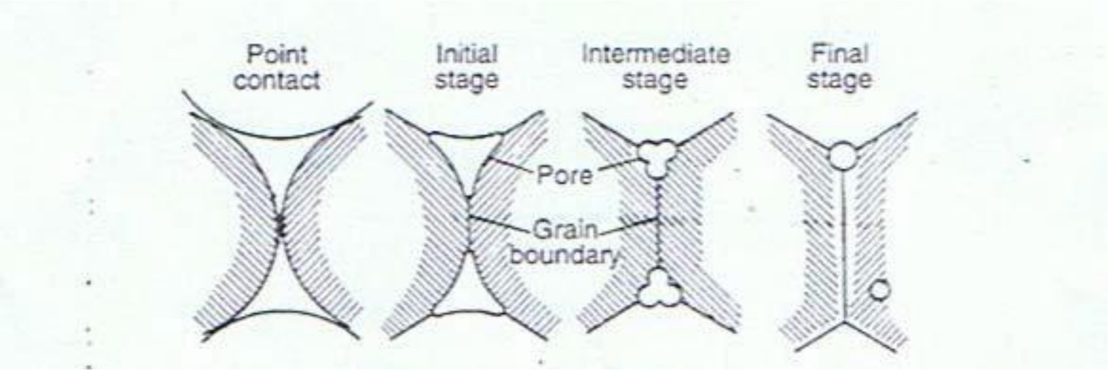


Fig. 2.5 shows various sintering stages [48]

Sintering of powder systems involving more than one component, in either elemental and/or master alloy powder form, is accomplished by interdiffusion of component elements. Simply, the driving force for this interdiffusion is related to the concentration gradients in the compacts, and the process involves the progressive reduction of these gradients with time and sintering temperature. The path by which homogenization occurs depends on the nature of phase diagram of the alloy system in question at the sintering temperature. Homogenization may involve the formation and subsequent dissolution of one or more intermediate phases where the sintering temperature is above the solidus temperature.

2.7 Mechanical properties of materials

Mechanical properties include those characteristics of material that describe it behavior under the action of external forces. The response of the materials to applied forces will depend on the type of bonding, the structural arrangement of atom or molecules and the type, the number of imperfections, which are always present in solids except in rare circumstances. In our study it was concentrated on some of these mechanical properties such as hardness and compressive strength [9].

2.7.1 Compressive strength

Compressive strength is the ability of a metal to withstand pressure acting on a giving plane. The compressive stress is [59]:

$$\sigma = F/A \quad \text{------------------}(2.4)$$

Where

F: load applied perpendicular to specimen crosses section.

A_0: cross sectional area (perpendicular to the force) before applications of the load.

The compressive strain

$$\varepsilon = \Delta L/L_0 \text{ -------------------(2.5)}$$

Where ΔL is change in length, L_0 is the original length.

In axial compression, a test piece becomes shorter and its cross section increases. Thus the characteristics measured in compressive tests are the same as in the tensile test, but with opposite sign (contraction instead of elongation and an increase of the cross section area instead of its reduction).

Accordingly, actual stresses in compression are smaller than the conventional stresses (in contrast to the tensile test). The elastic, the proportional, and the yield limit measured in compression coincide quite well with those obtained in tension.

2.8 Thermal conductivity properties

2.8.1 Introduction

Heat transfer is the transition of thermal energy from a heated item to a cooler item. As a result of the second law of thermodynamics. Where there is a temperature difference between objects in proximity, heat transfer between them can never be stopped; it can only be slowed down. Classical transfer of thermal energy occurs only through conduction, convection, radiation or any combination of these. In each case, the driving force for heat transfer is a difference of temperature [60].

2.8.2 Heat conduction of metals

The transfer of energy could be primarily by elastic impact as in fluids or by free electron diffusion as predominant in metals or phonon vibration as predominant in insulators. In other words, heat is transferred by conduction when adjacent atoms vibrate against one another, or as electrons move from atom to atom.

Conduction is greater in solids, where atoms are in constant close contact. In liquids (except liquid metals) and gases, the molecules are usually further apart, giving a lower chance of molecules colliding and passing on thermal energy. Vibrations of individual atoms in solids are not independent from each other. The coupling of atomic vibrations of adjacent atoms results in waves of atomic displacements. Each wave is characterized by its wavelength and frequency. For a wave of a given frequency ν, there is the smallest “quantum” of vibrational energy, hν, called phonon.

Thus, the thermal energy is the energy of all phonons (or all vibrational waves) present in the crystal at a given temperature. Metals such as aluminum are usually the best conductors of thermal energy. This is due to the way that metals are chemically bonded: metallic bonds (as opposed to covalent or ionic bonds) have free-moving electrons and form a crystalline structure, greatly aiding in the transfer of thermal energy [61].

2.8.3 Thermal conductivity of metals

Thermal conductivity is a material property that is primarily dependent on the medium's phase, temperature, density, and molecular bonding. This parameter is defined as the infinitesimal quantity of thermal energy, δQ, transmitted during an infinitesimal time interval dt through a thickness dx, in a direction normal to a surface of area (A), due to a temperature difference (dT) [62]:

$$\frac{\delta Q}{dt} \equiv q(x,t) = -\kappa A \frac{\partial T(x,t)}{\partial x} \quad \text{----------------(2.6)}$$

Where q is the so-called heat current. Eq. (2.6) is suitable to describe the heat transport along a specified direction (here denoted by x). In general, three-dimensional cases, the spatial variation of the temperature must be taken into account and the temperature gradient should replace its partial derivative in the right hand side of Eq. (2.6). Accordingly, the heat current will become a vector quantity. Eq. (2.6) is valid when the medium is homogeneous.

When the heat transport occurs in inhomogeneous media, the thermal conductivity varies with x.

Chapter Three

Experimental method and procedures

Chapter Three

Experimental method and procedures

3.1 Introduction

This chapter includes the experimental part that explain the types of materials which were used to prepare samples for tests, the apparatus used for each test and the methods that were followed.

3.2 Materials: -

3.2.1 Aluminum (Al): Aluminum is the most abundant metal in the earth's crust and it is widely distributed. It is a very reactive element and is never found as the free metal in nature. So it is found combined with other elements, most commonly with oxygen, silicon, and fluorine. These chemical compounds are commonly found in soil, minerals (e.g., sapphires, rubies, turquoise), rocks (especially igneous rocks), and clays. Aluminum as the metal is obtained from aluminum-containing minerals, primarily bauxite. It is used to make beverage cans, pots and pans, airplanes, siding and roofing, and foil. While powdered aluminum metal is often used in explosives and fireworks [63].

In its pure state, aluminum is, however, a relatively soft metal with a yield strength of only 34.5 N/mm^2 (5,000 lb/in^2), a tensile strength of 90 N/mm^2 (13,000 lb/in^2) and has a relatively high corrosion resistance through the development of a wide range of alloys, however, very varied strengths and ductility can be achieved, and this has led to the many applications of today [64].

Table 3.1. Explain the physical properties of aluminum [63]:

Chemical symbol	Al
Color	Silver
Density	2.7 g/cm^3
Melting point	660 ^{o}C
Purity	99.9 %

3.2.2 Spinel ($MgAL_2O_4$): magnesium aluminate spinel (MAS) possesses a unique combination of desirable properties such as high melting point(2135^{0}C), high strength at elevated temperature, good chemical inertness, low thermal expansion coefficient and good thermal shock resistance. $MgAl_2O_4$ became the significant choice in many structural, chemical and electrical applications [2]. Besides, it also used in many ceramic applications such as cement rotary kilns, lining of steel ladles, glass tank furnace and other ceramic application [7].

Table 3.2. Explain the physical properties of Spinel [7]:

Chemical symbol	$MgAl_2O_4$
Color	White
Density	3.5 g/cm^3
Melting point	2135 ^{o}C

3.3 Apparatus

3.3.1 Die description

Two cylindrical dies, the first with dimensions 12mm in diameter and 50mm height for forming the samples by semi-dry pressing to test physical, mechanical properties and microstructure, the second with dimensions 50mm in diameter X 90mm height for testing thermal conductivity of Spinel-Al samples as shown in Fig. 3.3, 3.4, 3.5 and 3.6 respectively.

Fig 3.3 The die used in compacting Spinel-Al samples

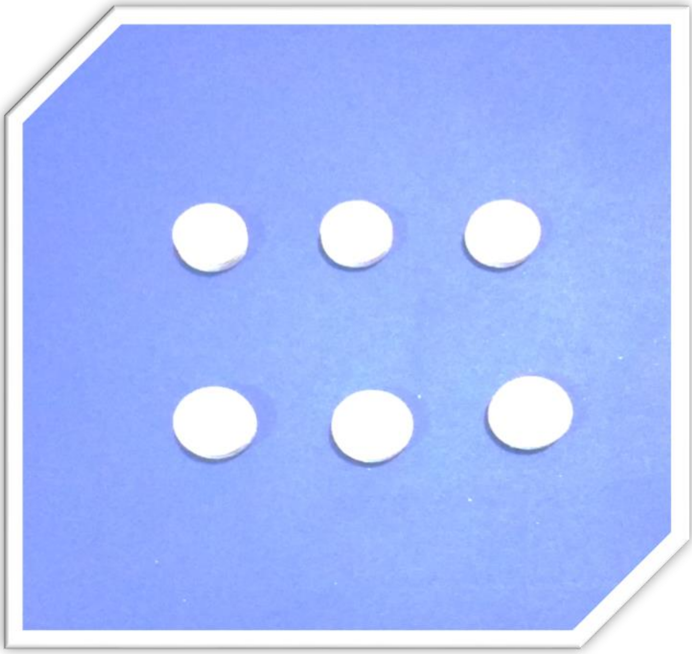


Fig 3.4 The compacted Spinel-Al samples

Fig 3.5 The die of Lees disk samples

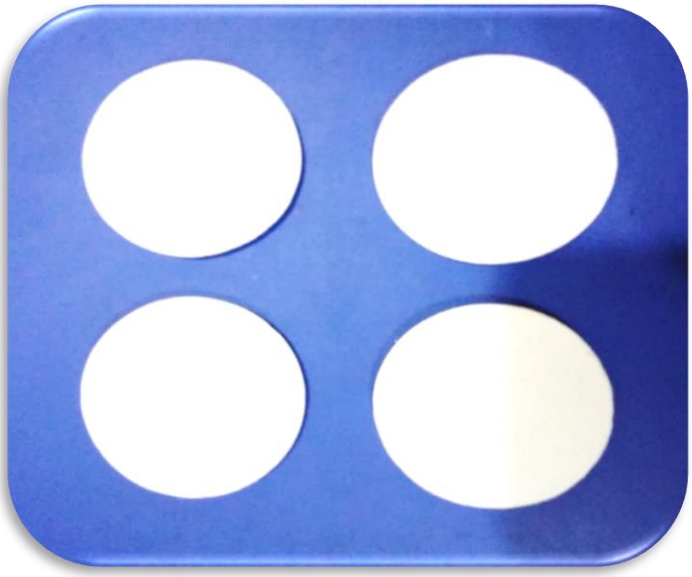


Fig 3.6 Lees disk samples of Spinel-Al composite

3.3.2 Sample preparation:

High pure aluminum Al (Central Drug House (P) Ltd. 7/28 Vardaan House, Daryaganj, New Delhi-110002 (India) purity 99.9 %) was used as a matrix material. As solid state synthesis is the conventional method to prepare the $MgAL_2O_4$ spinel via the reaction between MgO and Al_2O_3 as primary materials. This reaction achieved diffusion of Al^{+3} toward MgO and Mg^{+2} toward Al_2O_3

forming a spinel layer which acts as a barrier layer against the diffusion agents [65-67]. So synthesized stoichiometric $MgAL_2O_4$ spinel was prepared according to single stage firing process from an intimate mixture of its constituents (28wt % MgO +72wt% Al_2O_3) with 1:1 molar ratio heated at 1200 and 1400^0C for 2h was used as reinforced material to obtain Spinel-Al composite.

Generally, end product material depends essentially on some basic properties of the powders, such as high purity, chemical homogeneity, particle size and narrow size distribution. So, different preparation routs have been developed to obtain high quality powders which leading to an improvement in processing Spinel-Al composite [68].

After the presence of $MgAl_2O_4$ was confirmed, $MgAl_2O_4$ is added to the Al powder (starting material) with different weight percentages of $MgAl_2O_4$ Spinel (0, 2, 4, 6, and 8 wt.%), and then they are mixed by using ball milled for 5h.

Fig 3.7 The mixer of Spinel-Al powders

The powder mixtures were then semidry pressed in the form of pellets (12mm dia, x40mm height) using a hydraulic press under a pressure of (25 MPa) to get green specimens of composites of (12 mmx14 mm) dimension.

Fig 3.8 The hydraulic press for compacting spinel-Al samples

Spinel-Al composite samples were sintered at different temperatures ranging from 450-650 ^{0}C for 2h soaking time in an electric furnace (Nabertherm Company, Ltd., Germany).

Fig 3.9 The thermal furnace for sintering Spinel-Al samples

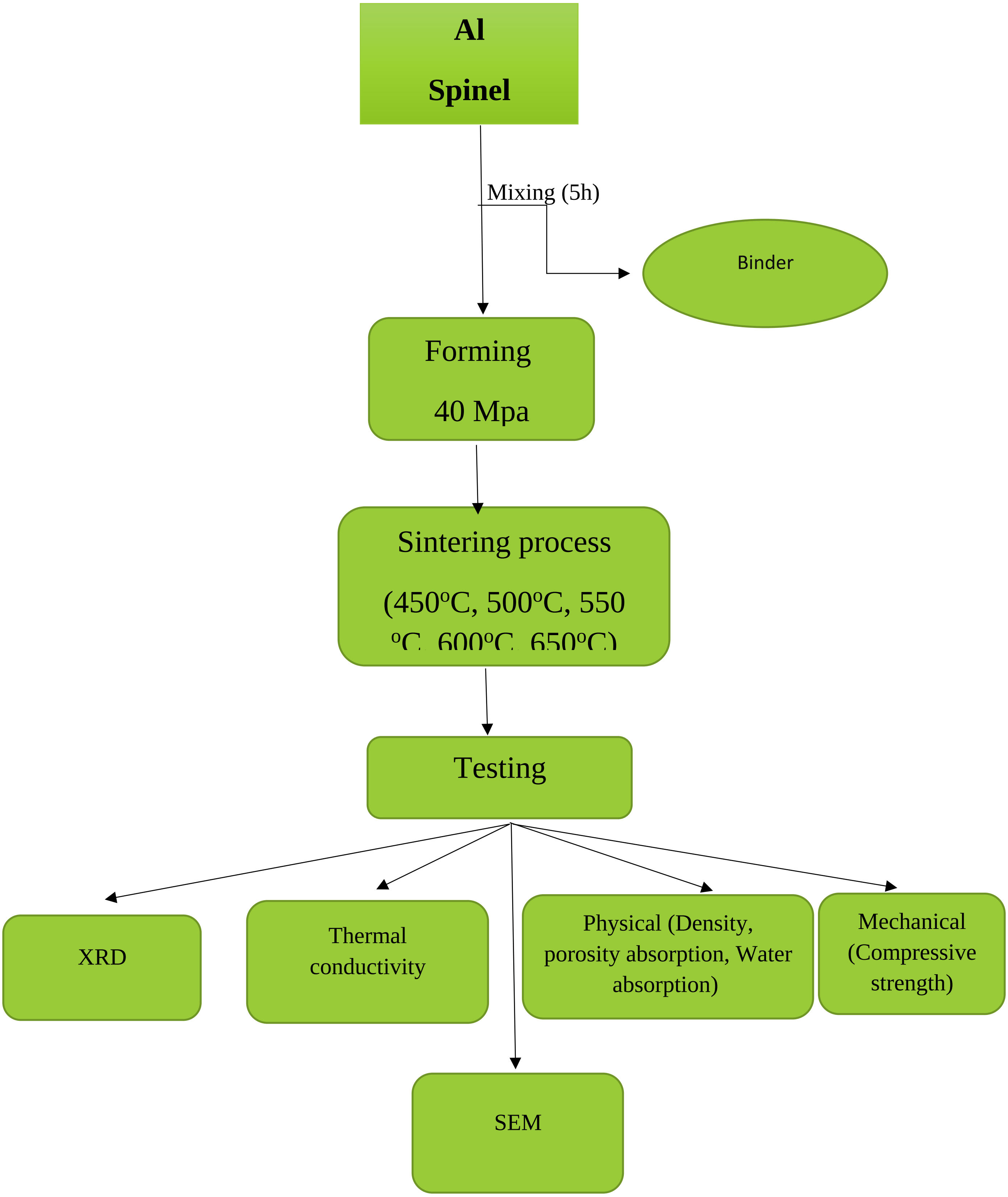


Fig. 3.10 A flow chart for processing Spinel-Al composite specimens

3.4 Test performed for Spinel-Al Composites

In this study, the most common method for investigation physical, mechanical and thermal properties to show the sintering behavior of Spinel-Al Composites as follows;

3.4.1 Density test:

The green and sintered densities of Spinel-Aluminum composite specimens may be obtained in terms of densities of its constituents by simple rule of composite, matrix and reinforced material densities [69].

$$\rho_c = \rho_r V_r + \rho_m V_m \text{ ------------ } (3.1)$$

Where:

ρ_c : Density of composite material

ρ_r : Density of reinforced material

ρ_m: Density of matrix material

V_r: Volume fraction of reinforced material

V_m: Volume fraction of matrix material

The conversions between weight fraction and volume fraction may be obtained from the following relation [70].

$$W_r = \rho_r / \rho_c V_r \text{ ----------------------- } (3.2)$$

$$W_m = \rho_m / \rho_c V_m \text{ ------------------- } (3.3)$$

Where:

W_r : Weight fraction of reinforced material

W_m : Weight fraction of matrix material

Varnier with accuracy of 0.01 mm was used to measure height and radius of the compacted specimens.

3.4.2 Water absorption

Apparent absorption of sintered samples was determined by the water displacement technique after applying the following equation [71]: -

$$\text{Water absorption W.A \%} = \frac{Ws - Wd}{Wd} \times 100 \quad \text{-------} \quad (3.4)$$

Where:

Ws: weight of sintered sample saturated in water (gm)

Wd: weight of dry sintered sample (gm)

3.4.3 Compressive strength

The compressive strength of the sinter samples was measured using an instron universal testing machine (Across International company (USA)) according to the following equation [71].

$$\text{Compressive Strength} = \frac{F}{A} \ (\text{Mpa}) \text{ ----------------- } (3.5)$$

Where:

F: Applied force (N)

A: cross section of sample (mm^2)

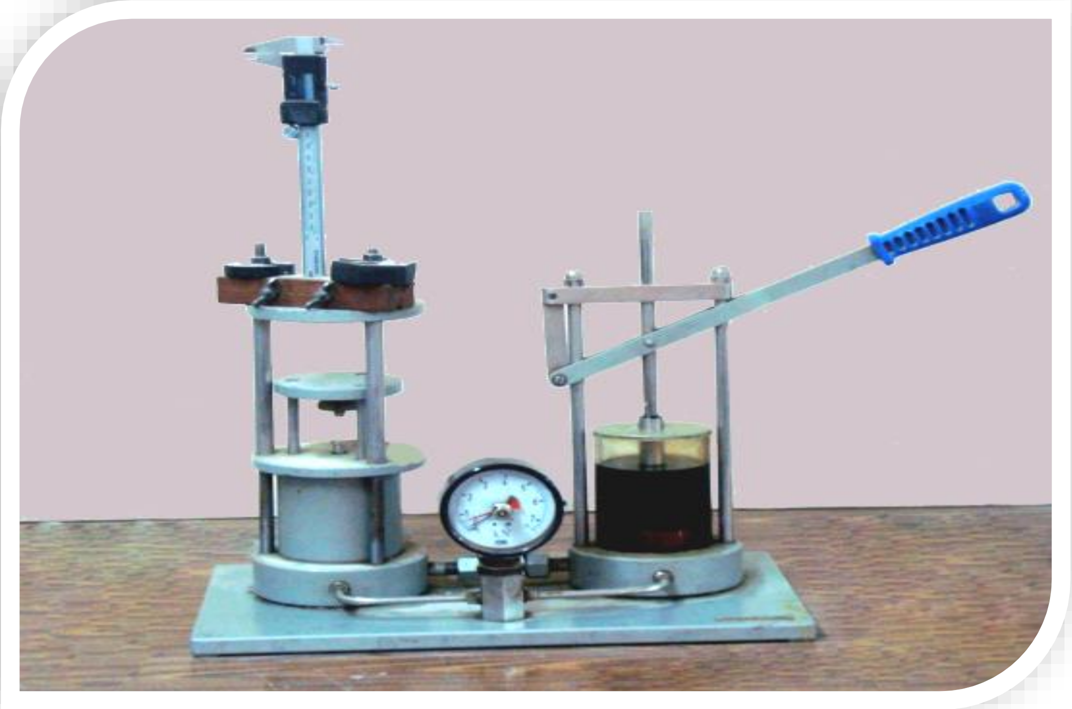


Fig 3.11 The compressive strength test devise

3.4.4 Thermal conductivity property

Thermal conductivity of a Spinel-Al composite in form of a disc can be determined by using Lee’s method. By applying Fourier’s Law, conductive heat transfer can be expressed as follow [72].

$$H = kA\,\frac{(T_2 - T_1)}{x} \quad \text{--------------} \quad (3.6)$$

Where:

H: The steady state rate of heat transfer

k: The thermal conductivity of the sample

A: The cross sectional area

$T_2 - T_1$: The temperature difference across the sample

X: Thickness of the sample

Generally speaking, there are a number of possibilities to measure thermal conductivity, each of them being suitable for a limited range of materials, depending on the thermal properties and the medium temperature. The most commonly used methods are Searle's method and Lee's disc method, for good and bad conductors of heat, respectively. In this study, we will use Lee's disc method to determine the thermal conductivity of a Spinel-Al composite.

Fig 3.12 Thermal conductivity device

3.4.5 Powder morphology and microstructure:

Powder morphology and microstructure study of Spinel-Al composites at firing temperatures 1200, 1400^{0}C were observed via scanning electron microscopy SEM and X-ray diffraction XRD.

3.4.5.1 X-ray Diffraction (XRD) test:

The crystalline phases and the crystallinity of the raw powders were examined by X-ray diffraction (D2 PHASER, Bruker Com.). All the measurement done in Baghdad University / Science College / Geologic Department. The phases and lattice parameter of the sintered bodies were examined by X-ray analysis using Si as an internal standard.

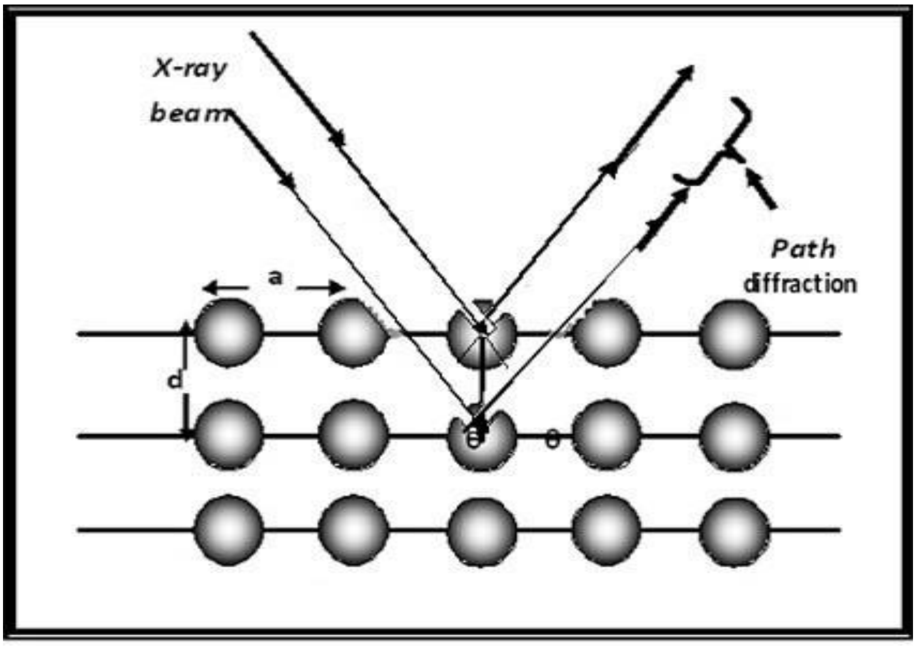


Fig 3.13 Braggs reflection on the crystal surface [73].

X-rays are electromagnetic radiation lying between ultraviolet and gamma rays in the electromagnetic spectrum. The wavelength of X-ray is expressed in angstrom units (A^0); 1 A^0 is equal to 10^{-8} cm.

Diffraction is a scattering phenomenon. When X-rays are incident on crystalline solids, they are scattered in all directions. In some of these directions, the scattered beams are completely in phase and reinforce one another to form the diffracted beams. Braggs law describes the conditions under which this would occur. It is assumed that a perfectly parallel and monochromatic X-ray beam, of wavelength λ, is incident on a crystalline sample at angle Θ [74].

$$n\lambda = 2\,d\,\sin\Theta \text{ -----------------(3.7)}$$

Where d= distance between the planes in the crystal, expressed in angstrom units, and n= order of reflection (an integer).

X-ray powder patterns can be obtained using either a camera or a powder diffractometer. Currently, diffractometers find widespread use in the analysis of pharmaceutical solids.

Fig 3.14 XRD device

3.4.5.2 Scanning electron microscope (SEM):

The micro structure of the sintered body was examined by SEM through observing the shape and size of Spinel-Al composite particles.

Scanning electron microscopy is used for inspecting topographies of specimens at very high magnifications using a piece of equipment called the scanning electron microscope. SEM magnifications can go to more than 300,000X but most semiconductor manufacturing applications require magnifications of less than 3,000 X only. SEM inspection is often used in the analysis of die/package cracks and fracture surfaces, bond failures, and physical defects on the die or package surface [75].

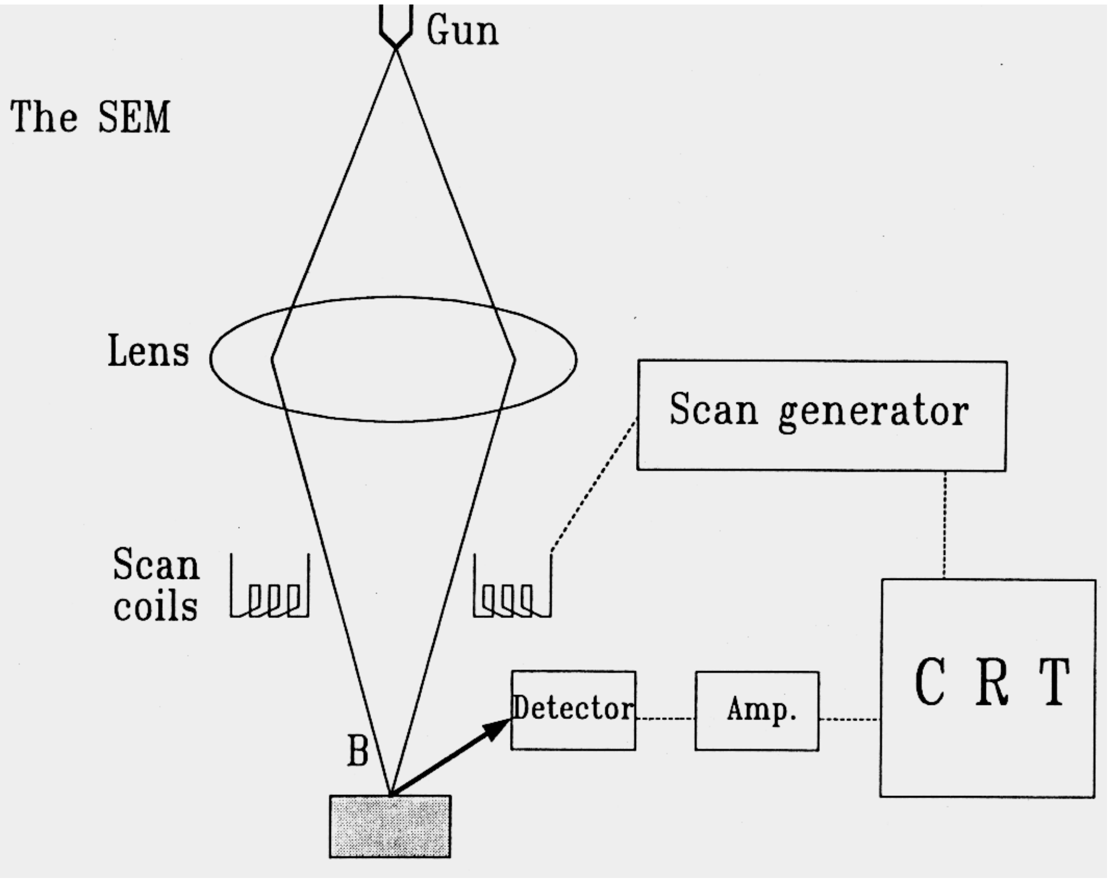


Fig 3.15 The SEM -scanning electron microscopy [73].

During SEM inspection, a beam of electrons is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attracted and collected by a positively biased grid or detector, and then translated into a signal.

To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed, and translated into images of the topography being inspected. Finally, the image is shown on a CRT.

The energy of the primary electrons determines the quantity of secondary electrons determines the quantity of secondary electrons collected during inspection. The emission of secondary electrons from the specimen increases as the energy of the primary electron beam increases, until a certain limit is reached. Beyond this limit, the collected secondary electrons diminish as the energy of the primary beam is increased, because the primary beam is already activating electrons deep below the surface of the specimen. Electrons coming from such depths usually recombine before reaching the surface for emission [76].

Aside from secondary electrons, the primary electron beam results in the emission of backscattered (or reflected) electrons from the specimen.

Backscattered electrons possess more energy than secondary electrons, and have a definite direction. As such they cannot be collected by a secondary electron detector, unless the detector is directly in their bath of travel. All emissions above 50 eV are considered to be backscattered.

Backscattered electron imaging is useful in distinguishing one material from another, since the yield of the collected backscattered electrons increases monotonically with the specimens atomic number. Backscatter imaging can distinguish elements with atomic number differences of at least 3, i.e., materials with atomic number differences of at least 3 would appear with good contrast on the image.

A SEM may be equipped with an EDX analysis system to enable it to perform compositional analysis on specimens. EDX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen [77].

Chapter Four

Results and Discussion

Chapter Four

Results and Discussion

4.1 Introduction

This chapter discussed the results of an attempt was made to explore the effect of stoichiometric $MgAl_2O_4$ produced via single firing stage at firing temperatures 1200,1400 ^{0}C as a reinforced material which added to the Aluminum (matrix material) on the physical, mechanical and thermal insulating properties of Spinel-Al composite.

4.2 XRD tests:

XRD differaction patterns of the spinel$_{1200^0C}$-Al samples fired at 650^{0}C are shown in Fig. 4.1. It can be seen that the strongest four peaks appeared at 2Ɵ values of 38.5^0, 44.67^0, 65.1^0 and 78.1^0. These peaks correspond to (111),(200) , (220) and (311) differaction planes of Al samples, whereas the strongest four peaks appeared at 2Ɵ values (38.7^0, 44.97^0, 65.3^0 and 60.19^0), (39^0, 45.2^0, 65.62^0 and 60.33^0), (38.9^0, 45.15^0, 65.47^0, and 60.4^0) and (38.85^0, 45.26^0, 65.6^0 and 60.4^0) corresponding to (111), (200), (220) and (333) for 2,4,6,8wt% of spinel respectively.

Intensity of the spinel-Al phase was found to be increase with increasing spinel content while Al intensity decreased may be due to the initiation of spinel reaction in the batch, which results in poorly crystalline spinel phase and lesser amount of reactant phases present.

Fig.4.2 XRD differaction patterns for the prodused spinel$_{1400^0C}$-Al samples fired at 650^{0}C, showed that the strongest four peaks appeared at 2ϴ values of 38.5^0, 44.67^0, 65.1^0 and 78.1^0. These peaks correspond to (111),(200) , (220) and (311) differaction planes of Al samples, whereas the strongest four peaks appeared at 2ϴ values of (38.65^0, 44.8^0, 65.1^0 and 60.12^0), (38.7^0, 44.9^0, 65.25^0 and 60.27^0), (39.16^0, 45.4^0, 65.71^0, and 60.34^0) and (38.81^0, 44.96^0, 65.45^0 and 59.66^0) corresponding to (111), (200), (220) and (333) for 2,4,6,8wt% of spinel respectively.

Fig. 4.1 and Fig. 4.2 confirmed that the growth rates as well as its high crystillanity of the prodused Spinel-Al composite depends on the firing temperature of the spinel which complete the spinallisation prosses [78]. Therefore, the advantage of our project is the synthesis of spinel-Al composite at low temperature of 650^{0}C.

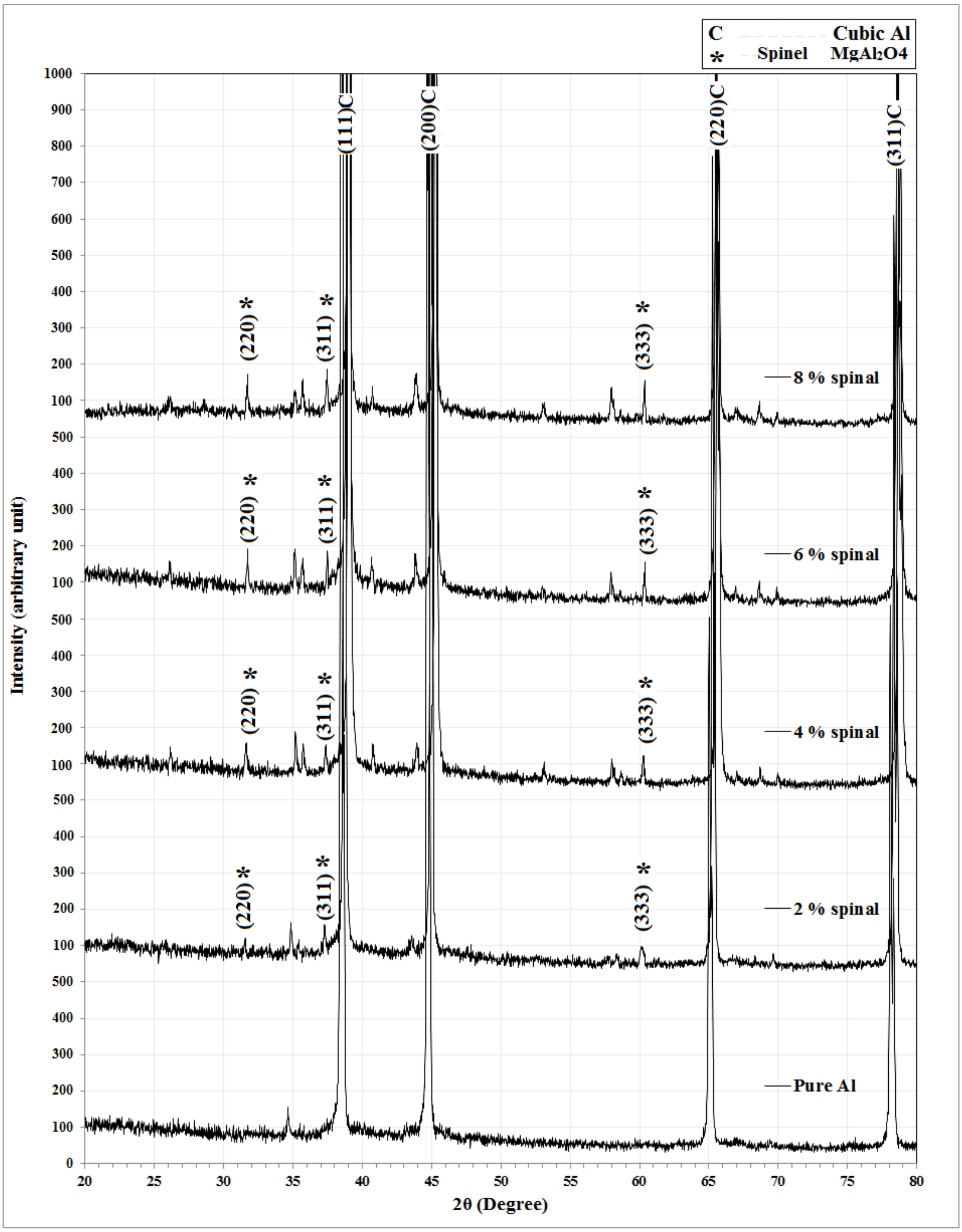


Fig. 4.1 XRD spectra of Spinel$_{1200^0C}$-Al composite samples fired at 650^{0}C with different spinel content.

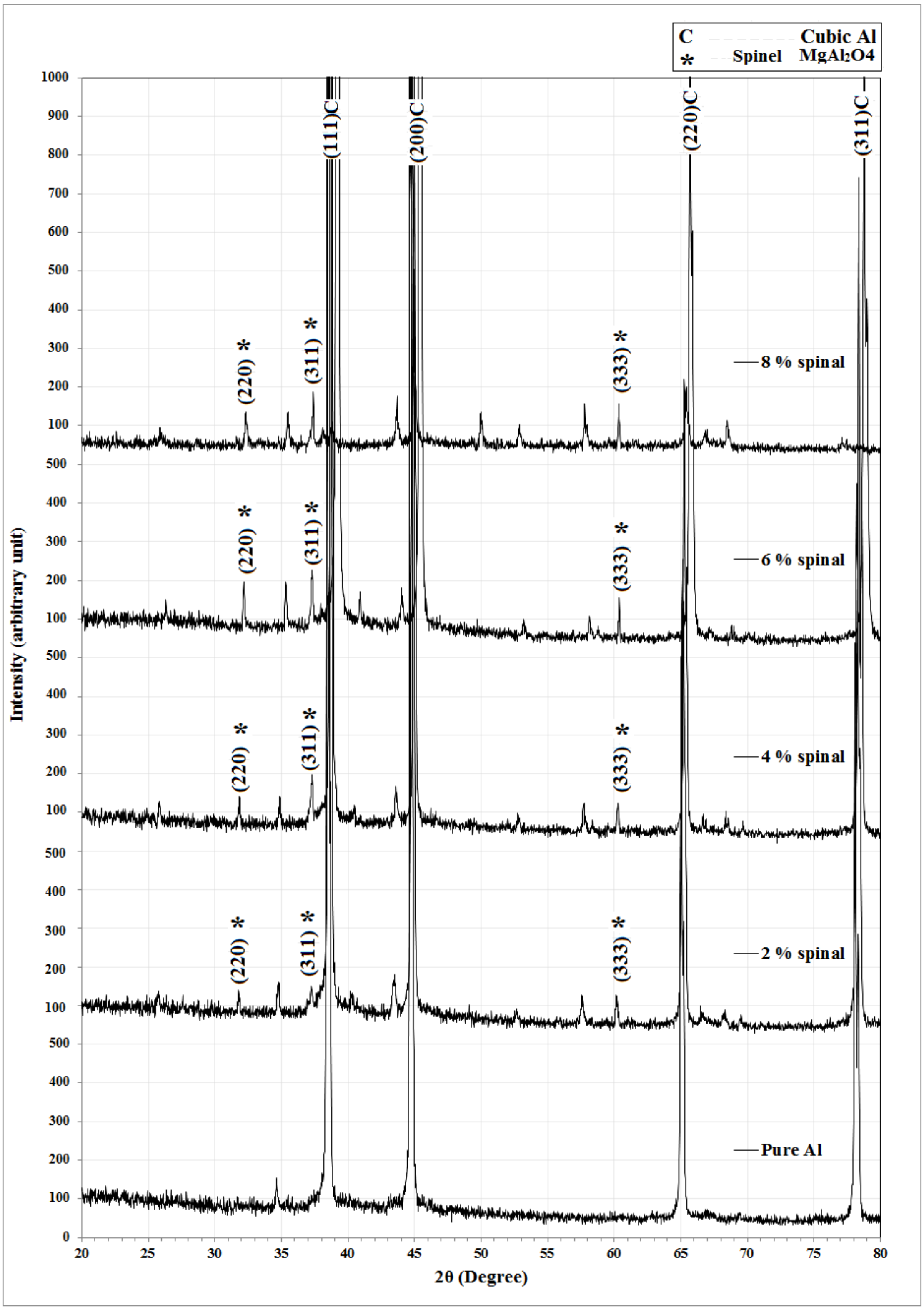


Fig. 4.2 XRD plot of Spinel$_{1400^0C}$-Al composite samples fired at 650^{0}C with different spinel content.

4.3 Microstructure tests:

Scanning electron microscope (SEM) is an instrument for observing and analyzing the surface microstructure of a bulk sample using a finely focused beam of energetic electrons.

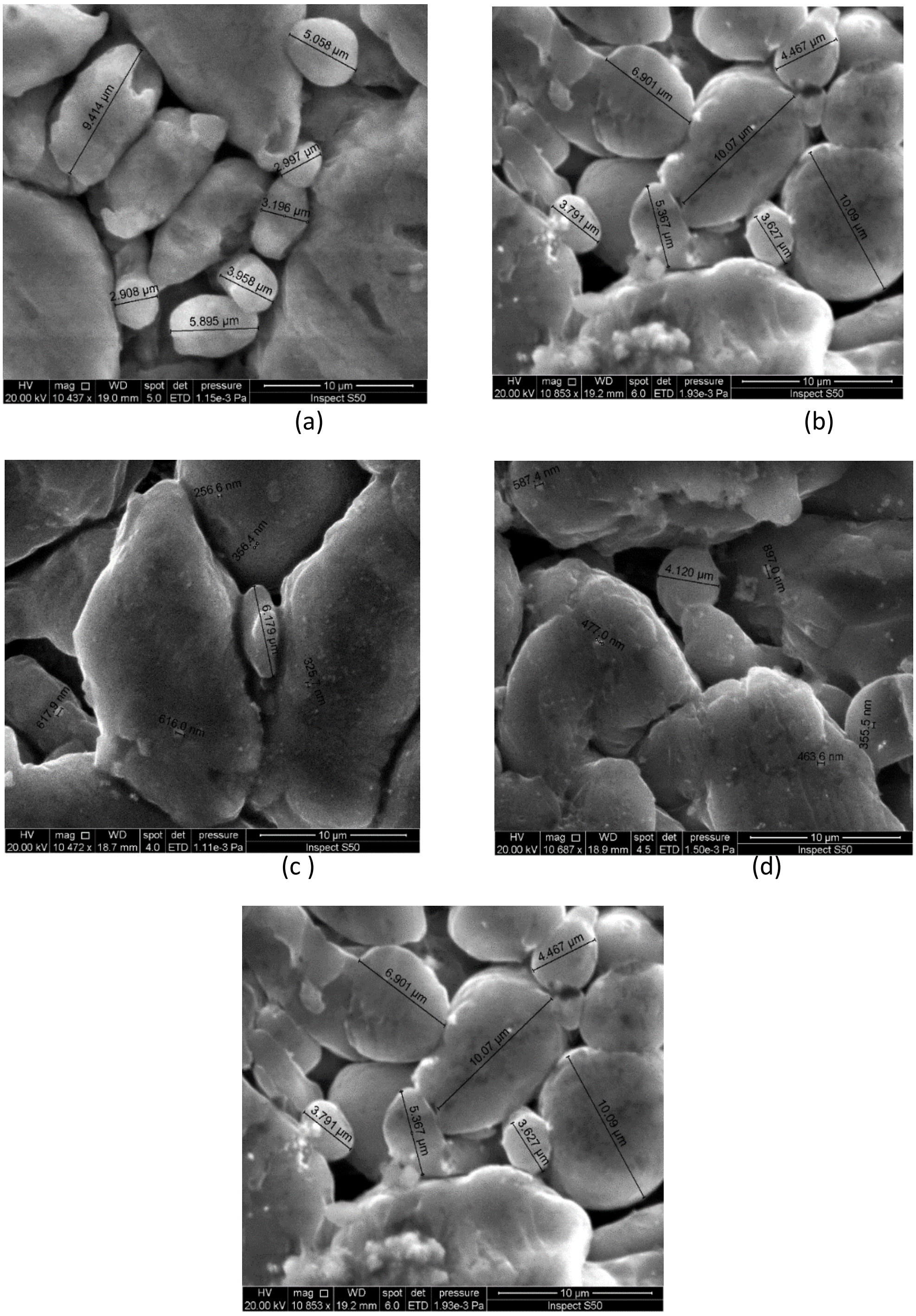
An electron-optical system is used to form the electron probe which may be scanned across the surface of the sample in a raster pattern.

Various signals are generated through the interaction of this beam with the sample. These signals may be collected or analyzed with the application of appropriate detectors. For imaging, the signal amplitude obtained at each position in the raster pattern may be assembled to form an image.

The morphological features of spinel$_{1200^0C}$–Al composites at 650^{0}C are shown in Fig. 4.3 (a, b, c, d, e). $MgAl_2O_4$ exhibit a good combination with Al matrix which improved the homogeneity and binding ability of reinforced spinel and Al matrix. So Particle agglomeration occurred during the dispersion process, and a lot of defects caused by the agglomeration were present in the composite, the coarse particles and the fine particles co-existed in the composite [44].

As can be seen, a large number of agglomerates are formed and their average grain size about 6.03, 6.17, 6.22, 6.3 (µm) for 2, 4, 6, 8 wt.% of $MgAl_2O_4$ respectively, whereas from Fig. 4.4 (a, b, c, d, e) their average grain size about 7.18, 7.57, 7.9, 8.48 (µm) for the same spinel content fired at 1400^{0}C respectively, where the particles spherical shape and the particle size increase due to the growth of particles with increasing calcination temperature of spinel from 1200 to 1400^{0}C [79]. Also, it has been notice that the agglomeration of Spinel$_{1200^0C}$-Al composite to be smaller than those of Spinel$_{1400^0C}$-Al composite as a result of better densification of these samples, but there was an abnormally growth aluminum grains.

Abnormal aluminum grain growth appeared only when the spinel content increased due to increasing the porosity and lowering heating rate.

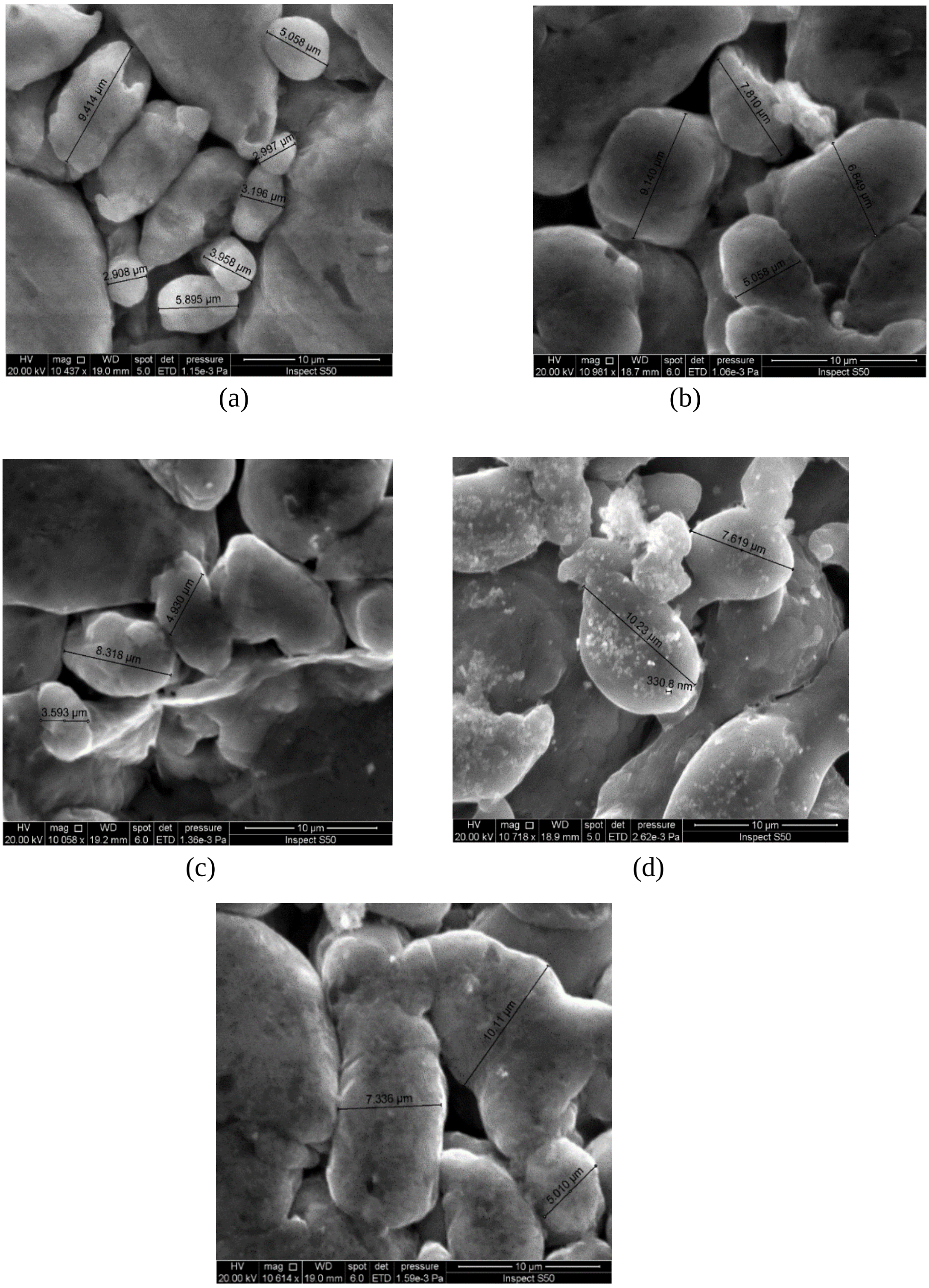


(a) (b)

(c) (d)

(e)

Fig. 4.3 Effect of Spinel content on microstructure of Spinel$_{1200^0C}$-Al composite sintered at 650^{0}C (a) 0wt%, (b)2 wt.%, (c) 4 wt.%, (d) 6 wt.%, (e) 8 wt.% Spinel content.



(a) (b)

(c) (d)

(e)

Fig. 4.4 Effect of Spinel content on microstructure of Spinel$_{1400^0C}$-Al composite sintered at 650^0C (a) 0wt%, (b)2 wt.%, (c) 4 wt.%, (d) 6 wt.%, (e) 8 wt.% Spinel content.

4.4 Green Density

Figs. 4.5,4.6 show the effect of spinel powders calcined at temperatures 1200, 1400 ^{0}C added with 0,2,4,6,8 wt.% to Aluminum on the green density of Spinel-Al samples. Any increase in spinel content resulted in increased green density of Spinel-Al composite.

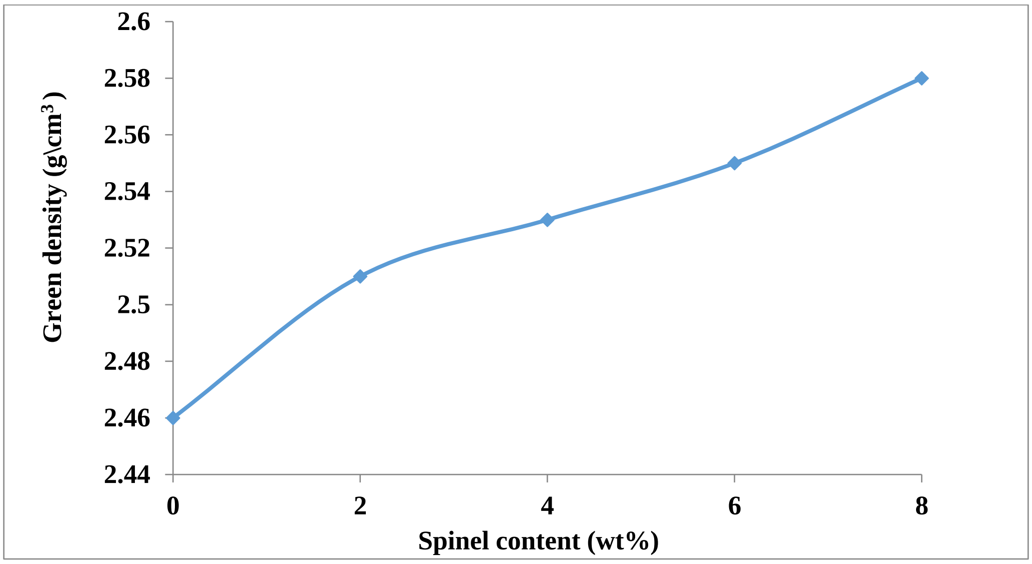


Fig.4.5 Effect of spinel content on the green density of spinel$_{1200}$^{0}C –Al composite.

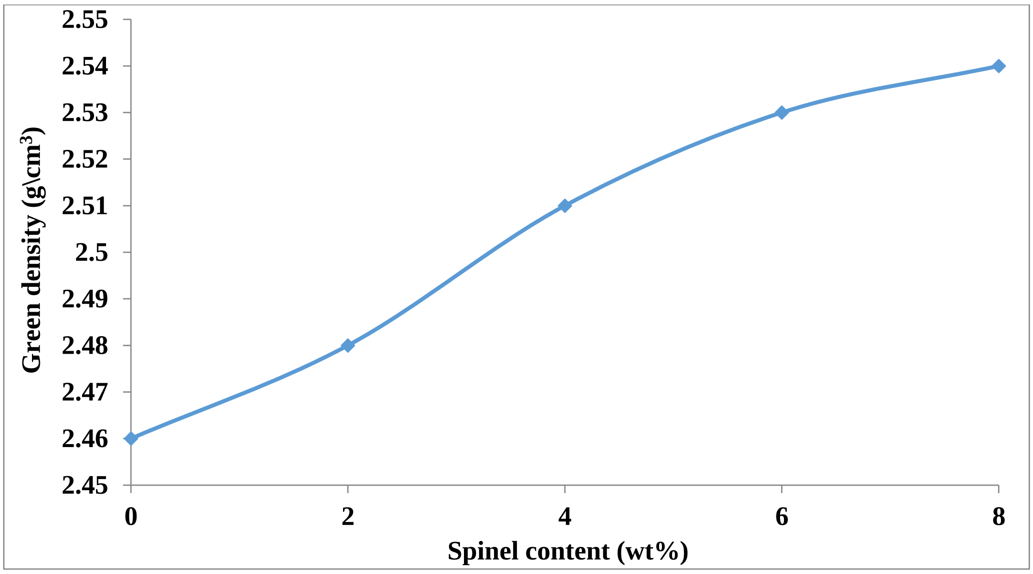


Fig.4.6 Effect of spinel content on the green density of spinel$_{1400}$^{0}C -Al composite.

4.5 Sintered Density

Figs. 4.7,4.8 show the effect of spinel content calcined at 1200 and 1400 ^{0}C, while Figs. 4.9,4.10 show the effect of firing temperature on the sintered density of spinel -Al composite. Sintered density of the samples increased with short range addition of spinel reaching their maxima at 2 wt.% of spinel addition and their densities decreased with long range addition of spinel content, due to the formation of a number of the pores and voids resulting from the consumption of Al matrix and Mg powder during sintering reaction. On the other hand, the network structure of the $MgAl_2O_4$ prevents the flow of metal and expansion of Al matrix at high temperatures [27].

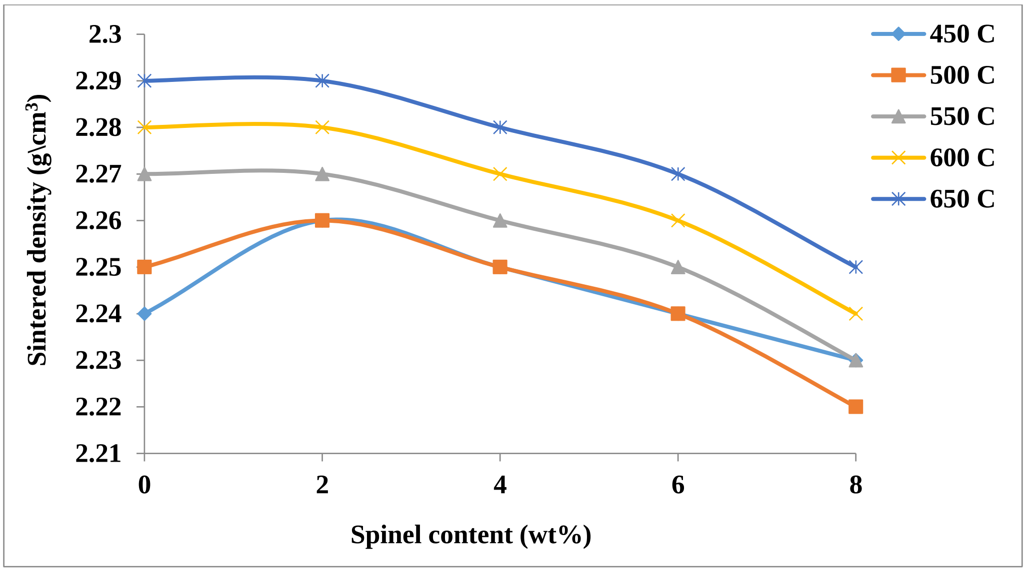


Fig.4.7 Effect of spinel content on the sintered density of spinel$_{1200}$^{0}C -Al composite.

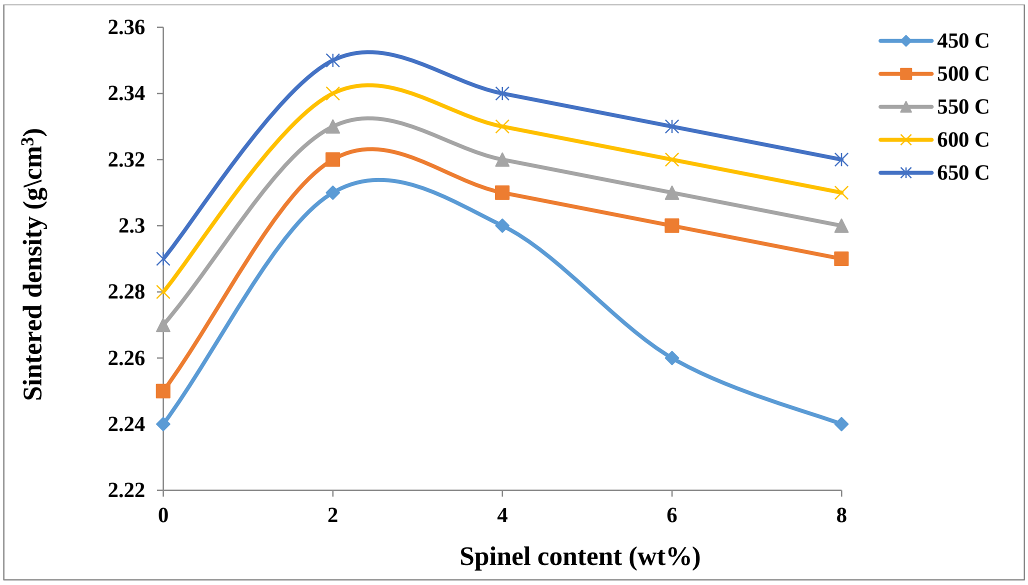


Fig.4.8 Effect of spinel content on the sintered density of spinel$_{1400}{}^{0}$C –Al composite.

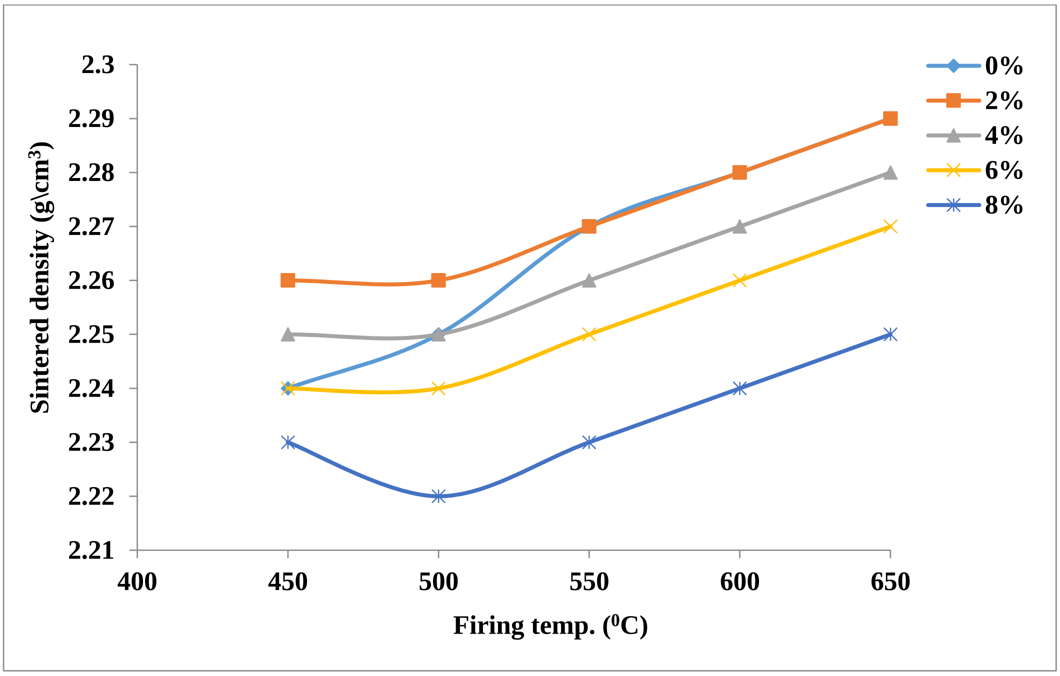


Fig.4.9 Effect of firing temperature on the sintered density of spinel$_{1200}{}^{0}$C –Al composite.

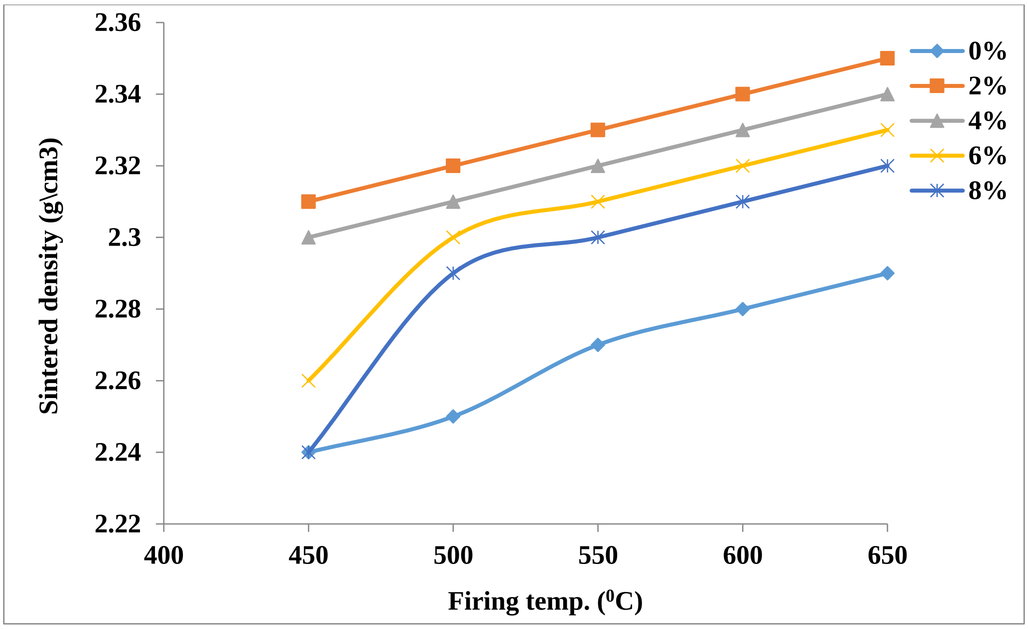


Fig.4.10 Effect of firing temperature on the sintered density of spinel$_{1400^0C}$–Al composite.

4.6 Water absorption

Figs. 4.11,4.12 and Figs. 4.13,4.14 show the effect of spinel content calcined at 1200 and 1400 ^{0}C and firing temperature on the water absorption of spinel -Al composite respectively. As a results of decreasing sintered density of Spinel-Al samples, water absorption of the samples increased with increasing spinel content while water absorption values are decreased as noted as a function of sintering temperature. It is interesting to note that 2 wt.% spinel is best to sinter Spinel-Al composite due to volume expansion associated with the spinel formation in both spinel firing temperature (1200 and 1400^{0}C) which cannot be condensed and solidificated of Spinel-Al composite bodies [80].

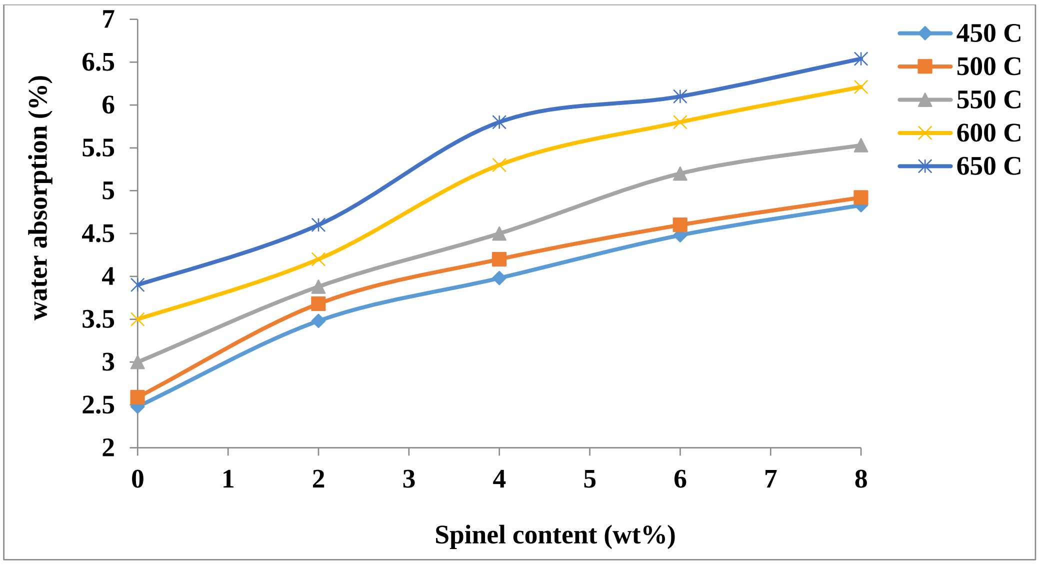


Fig. 4.11 Effect of spinel content on the water absorption of spinel$_{1200}$^{0}C –Al composite.

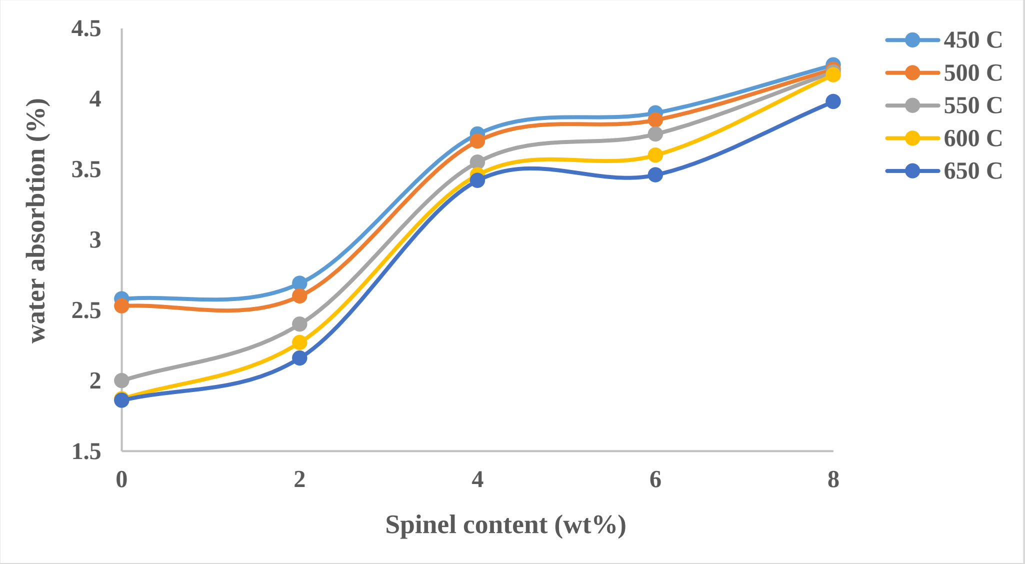


Fig.4.12 Effect of spinel content on the water absorption of spinel$_{1400}$^{0}C –Al composite.

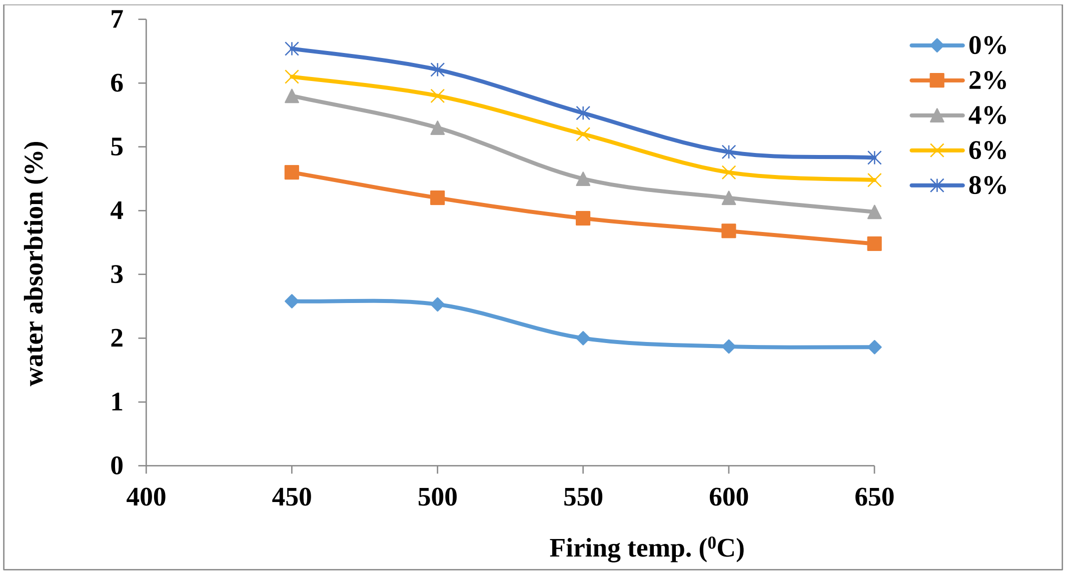


Fig.4.13 Effect of firing temperature on the water absorption of spinel$_{1200^0C}$ –Al composite.

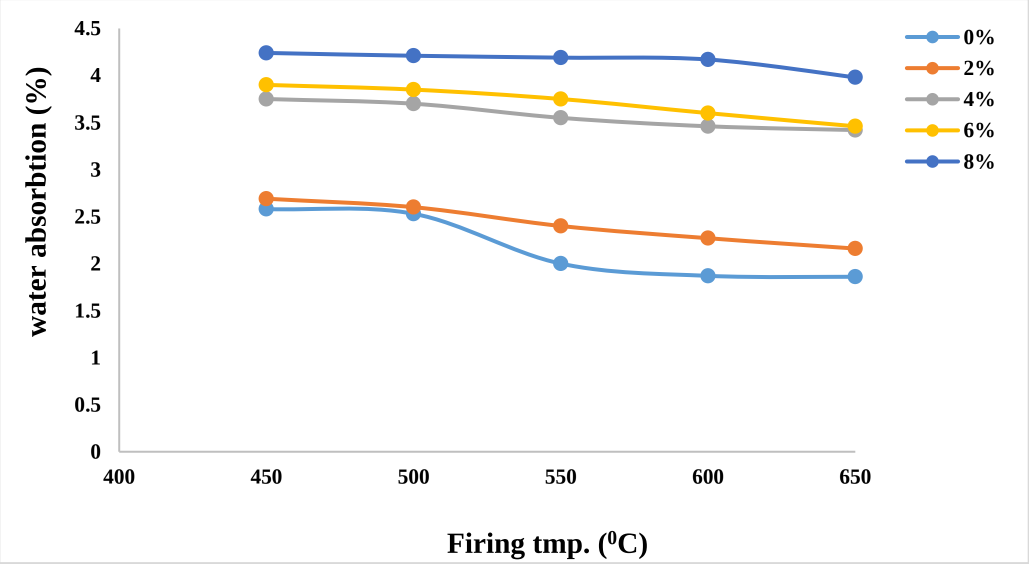


Fig.4.14 Effect of firing temperature on the water absorption of spinel$_{1400^0C}$ -Al composite

4.7 Compressive strength

To investigate the effects of spinel content on the compressive strength of the composites, compressive tests at firing temperature 650^{0}C were performed. It can be seen in Fig. 4.15 that when spinel content increased, the compressive strength of the composites decreases especially for spinel synthesis at 1400^{0}C firing temperature. According to Shen et al. [81], the compressive test is mainly different from the tensile test. So in spite of the generated spinel reinforcement is a kind of ceramic phase with high strength, and thus it can bear and transfer the extrinsic loads in the Al matrix. In general, compressive strength of Spinel-Al composite had lower compressive strength than Al material. In other words, compressive strength decreased gradually with increasing of spinel content in both cases (1200 and 1400 ^{0}C). Calcined temperature of spinel makes difference in compressive strength of Spinel-Al samples due to the rate of spinel formation. This related to the better sintering (densification) which accompanied by relatively lower contents of pores and voids [82].

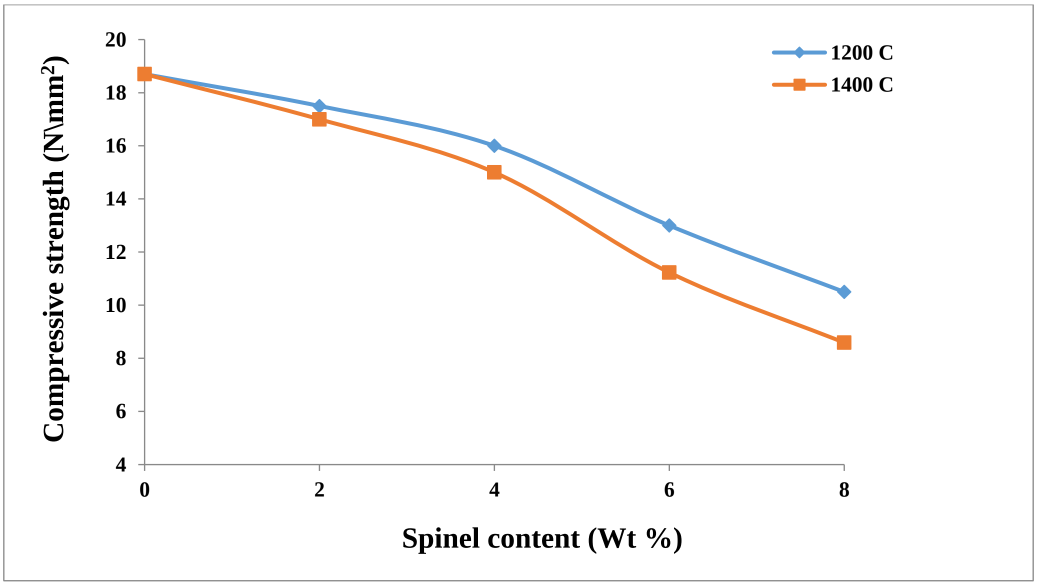


Fig.4.15 Effect of spinel content on the compressive strength of Spinel-Al composite fired at 650 ^{0}C

4.8 Thermal conductivity

Data on the thermal conductivity and other thermal properties of composite are of great engineering and physics significance. The amount of thermal conduction within a solid depends on the number of carriers which carry the heat energy and the degree of resistance to their movement within the solid [83]. The most important carriers are lattice vibrations (phonons). Thermal conductivity of

Spinel$_{1400^0C}$-Al composite samples fired at 650^{0}C was studied by static method, which finds the steady state thermal conductivity for 0, 2,4,6,8 wt.% of spinel content.

Thermal conductivity values of Spinel-Al composites against spinel content are plotted in Fig. 4.16. It is clearly observing that the composite samples retained less thermal conductivity compared to Al sample. This due to the effect of chemical composition of composite samples, its physical properties, and percentage impurities content of low softening temperature which causes weakness in thermal conductivity. So there is no obvious influence on the improvement of the thermal stability of the composite specimens.

In other side, with the increase of spinel content, a reticular structure of the spinel particles is formed to fix the Al matrix firmly. So, the Al matrix thermal conductivity can be strongly restrained [84].

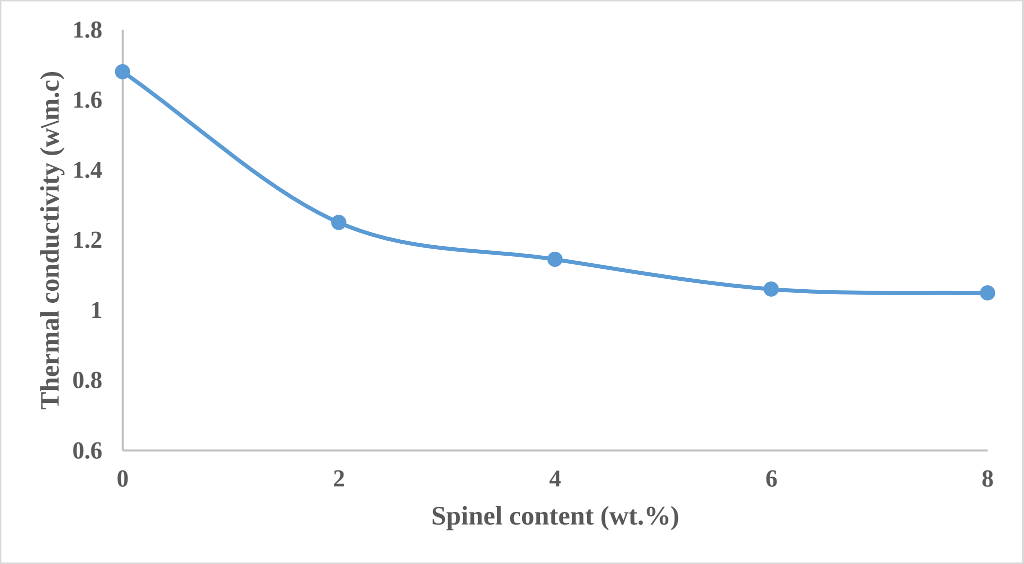


Fig 4.16 Effect of spinel content on the thermal conductivity of Spinel$_{1400^0C}$-Al composite at 650^{0}C

Chapter Five

Conclusion and Future work

Chapter Five

Conclusion and Future work

5.1 Conclusions

The main conclusions can be yielded from the obtained results of synthesis spinel-aluminum composites can be shown as follow:

1. The stoichiometric spinel-aluminum holds much promise for producing low-density composite material at relatively low temperature with acceptable physical, mechanical and thermal properties.

2. The conventional techniques (solid state reaction) for preparing spinel-aluminum composite materials with a different stoichiometric spinel contents are more complicated, so the full potential of the reinforcement to improve the overall properties of the composite cannot be exploited perfectly.

3. Spinel reinforced material is beneficial for the enhancement of the composite properties due to good combination between reinforced material particles and the matrix material particles.

4. The addition of reinforcements, such as spinel ceramic particles can apparently improve the overall properties of the composite which can extended the field of the desired uses of the Spinel-Al composites.

5. The sintering density, compressive strength, thermal conductivity, of the Spinel-Al composites decreased with increasing spinel content and firing temperature, whereas water absorption of spinel-Al composite increase with increasing spinel content and firing temperature.

6. 2 wt.% of spinel content is the best percentage weight which can be refer to added as a reinforced ceramic material to the aluminum matrix metal to improve the properties of the Spinel-Al composite.

5.2 Future work

1- Study the sintering behavior of spinel-aluminum composite prepared by one of the advance processing method such as sol-gel, precipitation, and hydrolysis techniques.

2- Study of the spinel-aluminum composite prepared from stoichiometric spinel reinforced material synthesis by two stage firing process and aluminum matrix material.

3- Study the effect of spinel whisker addition on the sintering behavior of spinel-aluminum composite.

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الخلاصة

تكتسب المواد المتراكبة نوع سبينل-المنيوم اهمية كبيرة لما تتمتع به من كثافة واطئة فضلا عن خواصها الفيزيائية والميكانيكية وعزل حراري جيد مقارنة بنظيراتها من المواد المتراكبة الاخرى. يهدف هذا البحث إلى دراسة احد الطرق التقنية التقليدية الاكثر شيوعا فضلا عن دراسة سلوك التلبيد للمادة المتراكبة سبينل-المنيوم من خلال تشخيص وتحديد الخصائص الفيزيائية والميكانيكية و حيود الاشعة السينية والبنية المجهرية لعينات المتراكب سبينل-المنيوم.

تم استخدام المغنيسيا (MgO) والألومينا (Al_2O_3) كمواد أولية لتحضير سبينل نوع الومينات المغنسيوم (MA) من خلال عملية حرق المواد الداخلة في تحضيره لمرحلة واحدة (تفاعل الحالة الصلبة).

تم إضافة السبنيل (مادة مدعمة) التي حضرت عند درجات حرارة 1200م^0 و 1400م^0 بزمن انضاج ساعتان وبنسب وزنية تتراوح بين 8,6,4,2 ٪، إلى الألومنيوم (مادة اساس) لتحضير المادة المتراكبة سبينل-المنيوم و في درجات حرارة تتراوح مابين 450-650م^0 عبر تفاعلات الحالة الصلبة (SSR). حيث تم دراسة وتشخيص كل من معاملات التلبيد، حيود الاشعة السينية، التركيب المجهري، الخصائص الميكانيكية والحرارية للمنتج المتراكب سبنيل-المنيوم، فضلا عن تأثير اضافة محتوى السبينل ودرجة حرارة التلبيد على سلوكية تلبيد المادة المتراكبة.

وقد أظهرت النتائج أن افضل كثافة تلبيد وامتصاصية الماء والانضغاطية فضلا عن الموصلية الحرارية للمنتج المتراكب كانت عند اضافة السبينل المحروق بدرجتي حرق 1200 و 1400م ^{0} بمحتوى 2 ٪ وبدرجة حرق المادة المتراكبة عند 650م ^{0}.

كما اظهرت دراسة حيود الأشعة السينية لعينات المادة المتراكبة سبينل-المنيوم، ان المنتج المتراكب يظهر اعلى القمم لعناصره عند القيم البلورية (38.7^0، 44.97^0، 65.3^0 و 60.19^0)، (39^0، 45.2^0، 65.62^0، 60.33^0)، (38.9^0، 45.15^0، 65.47^0 ، و 60.4^0) و (38.85^0، 45.26^0، 65.6^0، 60.4^0) والتي تتوافق مع مستوياته البلورية المتعددة وهي (111) و (200) و (220) و (333) وبنسب وزنية تتراوح 2،4،6،8 % من محتوى السبينل على التوالي.

وقد اظهرت نتائج دراستنا لمورفولوجيا المتراكب سبينل-المنيوم أن معدل الحجم الحبيبي يتراوح بين 6.03، 6.17، 6.22، 6.3 (ميكرون) لمحتوى السبينل وبنسبة وزنية 2، 4، 6، 8٪ على التوالي والمحروق بدرجة حرارة 1200م^0 وبزمن انضاج لمدة ساعتان ، في المقابل تراوح معدل الحجم الحبيبي

ما بين 7.18، 7.57، 7.9، 8.48 (ميكرون) على التوالي ولنفس النسب الوزنية من محتوى السبينل والمحروق بدرجة حرارة 1400م^0 ولنفس فترة الانضاج.

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للمتراكب $MgAl_20_4$-Al

رسالة
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كجزء من متطلبات نيل درجة ماجستير في علوم الفيزياء

من قبل

علي حسين صالح
بكالوريوس جامعة النهرين 2014

بأشراف

أ.م.د. قاصد عبدالستار صالح الجنابي

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