STUDY OF ALUMINA GRAPHITE COMPOSITE USING POWDER METALLURGY PROCESSING TECHNIQUE

by

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A thesis submitted in fulfillment of the requirements for the degree of Bachelor of Applied Science (Materials Technology)

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DECLARATION

I hereby declare that the work embodied in this Report is the result of the original research and has not been submitted for a higher degree to any universities or institutions.

_____

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I certify that the Report of this final year project entitled "study of the alumina graphite composite using powder metallurgy processing technique" by Suliana Binti Ab Razak, matric number E13A320 has been examined and all the correction recommended by examiners have been done for the degree of Bachelor of Applied Science (Materials Technology), Faculty of Earth Science, University Malaysia Kelantan.

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Study of Alumina Graphite Composite using Powder Metallurgy Processing Technique

ABSTRACT

The alumina graphite composite were successfully prepared by powder metallurgy technique. Through this method low energy milling machine was used. The effect of the microstructural and compaction were determined according to different milling time in this study. The different milling time that involved were 15, 30, 45 and 60 h and the different compaction pressure were 200, 400, 600 and 800 MPa. Properties of the milled powders covered a green density results follow up with densification parameter. As the high milling time, the pattern of the graphite and alumina diminished and become wider due to the well homogenizing powders during milling. Crystallite size decrease due to high milling time and the optimum milling time was determined at 45 h. Microstructure of the milled powders with different hours of milling resulting the changing size of particle become smaller, finer grain size produced as higher time of milling powders.

Kajian Komposit Alumina Graphite Menggunakan Serbuk Logam

ABSTRAK

Alumina grafit komposit telah disediakan dengan menggunakan teknik serbuk logam. Jenis mesin yang digunakan ialah pengisar jenis berkuasa rendah. Dalam kajian ini, kesan kepada mikrostruktur dan mampatan ditentukan mengikut masa kisaran yang dijalankan secara berbeza-beza. Masa yang digunakan dalam kajian ini adalah pada 15, 30, 45 dan 60 jam, manakala mampatan yang dikenakan untuk setiap serbuk yang terkisar ialah 200, 400, 600 dan 800. Semakin lama masa yang diambil untuk mengisar serbuk, semakin hilang atau mendatar puncak grafit dan alumina di dalam corak XRD disebabkan oleh gaulan yang sekata semasa kisaran berlaku. Saiz kristal semakin rendah disebabkan oleh tempoh masa yang lama semasa kisaran dan dapatan menunjukkan 45 jam masa untuk kisaran serbuk adalah yang optimum. Mikrostruktur serbuk yang dikisar dengan menggunakan tempoh masa yang berbeza dan semakin lama masa yang diambil menghasilkan perubahan saiz zarah menjadi semakin kecil dan halus.

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LIST OF ABBREVIATIONS

CMC	Ceramic Matrix Composite
MMC	Metal Matrix Composite
PMC	Polymer Matrix Composite
PM	Powder Metallurgy
XRD	X-Ray Diffraction
OM	Optical Microscope
Al_2O_3	Alumina
MGP	Mesophase Graphite
nm	Nanometer
WH	William Son

CHAPTER 1

INTRODUCTION

1.1 Background

A composite material is typical invented in engineering materials to improve the performance for better life. Composite is defined as two or more chemically distinct materials which combined to improve properties over individual material. This material was produced with two medium which so called reinforcement and matrix. Reinforcement provide structural properties to the composite while the matrix as a binder for a composite (Banga et al., 2015). This is also having various classification of composite such as laminate and sandwich. This material leads to strong, lower corrosion rate, lightweight and more flexibility in design.

Composite have been classified for several types such as ceramic matrix composite (CMC), metal matrix composite (MMC) and polymer matrix composite (PMC) (Vijayaraghavan, 2007). This type was combined with matrix that based on ceramic, metal and polymer for different applications. Generally, CMC used in structural application like used in part rocket and jet engine. While, for MMC was used in semiconductor industry, aerospace and automotive. On the other hand, for PMC it was commonly used in aerospace and automotive regarding the particularly lightweight properties (Hussain et al., 2006).

CMC was invented to rid the weakness in reliability of monolithic ceramics and intrinsic brittleness. CMC provide a better characteristics in high temperature stability, high thermal shock resistance, hardness, non-corrosion and lightweight (Vijayaraghavan, 2007). Although ceramics widely used in engineering materials, but it consists of drawbacks in mechanical properties and in inherent brittleness (Galusek and Galusková, 2015).

Alumina (Al_2O_3) is classified in group three element of aluminium oxide. It is known as very stable and robust material. In addition, it can be produce in high mass production due to cheap compare with other materials like titania, zirconia . Al_2O_3 is made from aluminium which is oxidized become aluminium oxide. Al_2O_3 is electric insulator but high thermal conductivity. Al_2O_3 ceramic is widely used in various application such as electrical insulator, refractory materials and wear resistance materials (Lee, 2014). This due to its properties which is high hardness, excellent dielectric properties and good thermal properties (Kumari, 2013).

According to Geric (2010), Al_2O_3 has high relative density and contains less than 2% porosity, hardness of 1600HV, which 16Gpa Knoop. Al_2O_3 is famous with mechanical and chemical properties but have lack in brittleness. Thus, it has limitations in design due to intrinsic stiff and brittle characteristics.

Graphite is one of the naturally mineral carbons. It is highly conductive properties material, and it is used for high temperature application due to stable with high temperature. In fact, graphite is also very stable structure. Mesophase graphite (MGP) is graphitic carbon that used in various applications due to its stable structure of graphite and stable cycling performance (Fang et al., 2006). This material currently use in coating composite, lithium ion battery because of its properties of low in cost and potential plateau with acceptable capacity (Baptista et al., 2016).

Powder metallurgy (PM) is the branch of metallurgy dealing with metal and non-metal powders and then using them for economical manufacturing of high precision components. It also defines as processing of metal powders which is including the fabrication, characterization and conversion. Term of powder should be understood before dealing deeply with powder metallurgy. Initially, powder is known as finely divided solid and it can be metallic combined with other phases like ceramic or polymers. It characteristics was high in surface area to volume ratio (Corrochano et al., 2011).

In PM, the composite widely produce through this way is low in cost, able to come out with high volume fraction composite and extreme productivity plus feasible to fabricate components with complex geometry. The steps of processing of powder metallurgy consist of powder production, powder conditioning, powder compacting, sintering, sizing and testing or inspection (Sabzevari et al., 2015; Wang et al., 2016).

1.2 Problem Statement

The previous research on ceramic matrix composite based alumina has been conducted by other researchers come out with excellent result on some characteristics and coped with applications needed in world materials technology. However, this Al_2O_3 have limitation which is needs other materials to cooperate with the material system to make the better properties and enhance the characteristics of the composite. This material cannot stand alone because it will prone to failure.

In order to overcome the problems, alumina graphite composite was invented to produce low cost preparation. It is good in having reinforcement to enhance Al_2O_3 based composite properties. The characteristics of Al_2O_3 composite-embedded with graphite will be potentially give the promising and better alternative modification of the system.

1.3 Objectives

The objectives of this research are:

i. To determine the effect of milling time on structural and microstructural properties of Al_2O_3–graphite composite.
ii. To observe the effect of compaction pressure on Al_2O_3–graphite composite.

1.4 Scope of Study

This research study was divided into two parts. The initial part is to determine the effect of milling time on structural and microstructural properties of Al_2O_3–MGP composite. After that, the second part is to observe the effect of compaction pressure on Al_2O_3–MGP composite.

For the sample preparation, the composition of Al_2O_3 (>99.9% purity, average particle size >20 μm), and MGP with the composition of FMGP-A-D 50 ~ approximately size of 11 micron will be used in this study to prepare Al_2O_3–MGP composite. This sample will undergo the different parameter of milling time and the compaction pressure to be analysed.

CHAPTER 2

LITERATURE REVIEW

2.1 Composite

Composite is well-known as a material which tailored properties from two or more different materials that give the improvement of the properties (Prabu et al., 2016). Composite also define as compound materials which is different from alloys, the individual components retains their characteristics and it is homogeneous material on microscopic scale. Besides, the term which is easily to understand according to this type of material is a combination of two or more materials that called the reinforcing phase is in form of fibers, sheets, or particles and embedded in the other materials called matrix phase can be metal, ceramic and polymer (Vijayaraghavan, 2007).

Basically, this material was divide into two phase which is called continuous and discontinuous phase or it was well known by continuous for ‘matrix’ and discontinuous for ‘reinforcement’ (Banga et al., 2015). In composite, continuous fibers used in structural requests because of their outstanding mechanical strength (Cho et al., 2007). Matrix is one of the materials that function to become a prevention platform to formation of new surface flaws and abrasion. It also can be called as bridge to hold the other material used in composite. While, reinforcement provides structural properties to the composite in performance of stiffness, strength, thermal stability and load carried and increasing in mechanical properties (Banga et al., 2015).

Properties of the composite are according to their distribution, the properties of the matrix and the reinforcement itself that resulting in better properties. Composite have three types of classification which is polymer based composite, metal based composite and ceramic based composite (Hussain et al., 2006; Hussainova et al., 2016).

Composite is a material that meets a global demand for lightweight, high performance, environmental friendly, wear and corrosion resistant. The uses of composite widely used in various applications such as aerospace, automotive, home furniture, and cookware.

2.1.1 Polymer Matrix Composite

Polymer matrix composite (PMC) is classified when the polymer material used as a matrix. PMC also composed of matrix from thermoset like unsaturated polyester, epoxy or thermoplastic materials which is polypropylene (PP), polyethylene (PE) and embedded in the glass carbon, steel or Kevlar fibers (Banga et al., 2015). PMC have gained some factors which are in low rate of purchasing and unpretentious fabrication methods. It was suitable commercial in aerospace appliance due to its lightweight properties (Hussain et al., 2006).

Other properties that claim with this material are high in tensile strength, stiffness, fracture toughness, and also good resistant in abrasion, puncture and corrosion was discuss (Vijayaraghavan, 2007). Polymer composite widely used in making of the stuffs relating with sports, aerospace components and in automobile due to its reducing in weight (Hussain et al., 2006). Polymer materials are also used as reinforcement in

metal matric composite the behaviour of strain and stress alike with metal but difference in mechanical properties.

2.1.2 Metal Matrix Composite

Metallic materials used in this type of composite, such as aluminium, zinc, and copper. Metal matrix composite (MMC) have their own special properties due to its high specific stiffness, strength and wear resistant (Estrada et al., 2013). The fabrication of MMC is great due to its excellent properties of combination with their alloys that resulted in high specific strength, wear resistance and the particle reinforced MMC can be produced by powder metallurgy and melt processing (Li et al., 2008).

MMC widely used in automobile applications due to its properties that enhance its functions well in this field of industry which is making pistons, brake drum, and cylinder block because of great corrosion and wear resistance (Radhika et al., 2011). From the previous research, found that although this material is quite interesting in various applications but the fabrication of this material have to face some barriers according to their porosity formation issue, poor wettability and improper distribution of reinforcement (Prabu et al., 2016).

2.1.3 Ceramic Matrix Composite

Ceramic matric composite (CMC) is the material that embedded was from ceramic group and the common matrices is alumina and silicon nitride (Yongli, 2006) and other materials as reinforcement either metals, polymers or ceramics. In fact, combination of materials used knows as hybrid composite. CMC also define as reinforcing ceramic or metal phases with a ceramic matrix. Ceramic is known as brittleness but lack in reliability but this material have high tensile strength, high compressive strength, high young modulus and low density at temperature up to 1500 ºC (Lamon, 2011).

According to Aigbodion et al. (2010), CMC is widely used in application that relate with high temperature for example in jet engine, heat shield for space vehicles, aircraft brakes and heat treatment furnace. Elements that used in CMC for high temperature are usually silicon carbide, carbon, alumina, silica and zirconia. Ceramic also used as reinforcement agents because of their hardness, chemical inertness, and suitable physical properties (Estrada-Guel et al., 2013). Another properties that make this CMC used in engineering field is high temperature stability, good wear performance and high hardness (Hussainova et al., 2016).

Fibers as a reinforcement in the CMC also has been discussed that the optimum type of material is based on carbon fibers due to its very good cost performance ratio, stable under non-oxidizing conditions while polymeric and organic material not suitable in making this product because of their properties which have a degradation at temperature below 500 ºC including conventional glass fibers below 700ºC (Clau, 2008). The applications or uses of this type of ceramic usually in seals rings, laser tube, electric insulators and thermocouple tubes (Hussainova et al., 2016).

2.2 Alumina

A word of alumina (Al_2O_3) produced by the aluminium oxide which is from aluminium and oxygen with physical appearance of white colour or colourless crystalline substances. It is very stable material and have strong ionic interatomic bonding that make it fulfil the material characteristics. This material is made from bauxite, which ore that have water contain and naturally occurred. Bauxite processing come out with several steps before Al_2O_3 was produced (Gow and Lozej, 1993).

Al_2O_3 is also known as popular material in ceramic that used in advance ceramic and also as a one mixed material in composite making. This material have a great property that makes it is suitable in composite field for making the high properties composite products for certain applications (Omrani et al., 2016).

The structural of Al_2O_3 was describing by Auerkari, (1996) that have allotropic and oxygen ions packed in close packed hexagonal arrangement with aluminium ions called internal crystal structure and it is also does not depart from stoichiometry but impurities can greatly impact high temperature diffusion rate.

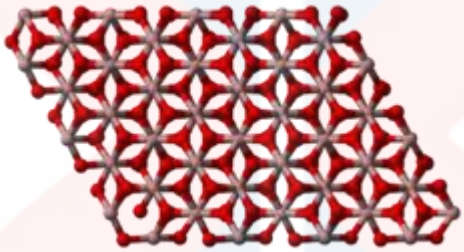

Figure 2.1 Al_2O_3 structure (Kumari, 2013)

2.2.1 Properties of Alumina

Desired properties make material become very precious and synthesized in every types of material according the uses. There are many material that has been revealed their good and desired properties that can brings the new chapter of the increasing rate of advance materials that give a beneficial for high technology applications. In fact, Al_2O_3 have special identities that make it very useful like low density, high hardness, toughness, and thermal stability (Mishra et al., 2014).

Besides, this material is cheap in purchase and abundance ability, it is also have others properties such as electrical insulator, chemical stability and melting temperature reached at 2040 ºC (Auerkari, 1996). In the scope of physical properties of Al_2O_3, it was divided in different type of group for example thermal and electrical properties. For

thermal conductivity of this material is in the range of 15-40 W/mK and electrical relative permittivity is 7.1-10.5 κ' (Auerkari, 1996). Figure 2.2 shows the example results or the pattern of XRD analysis of Al_2O_3. From the pattern, it can be observed that this material exist in crystalline phases as the formation of high peaks.

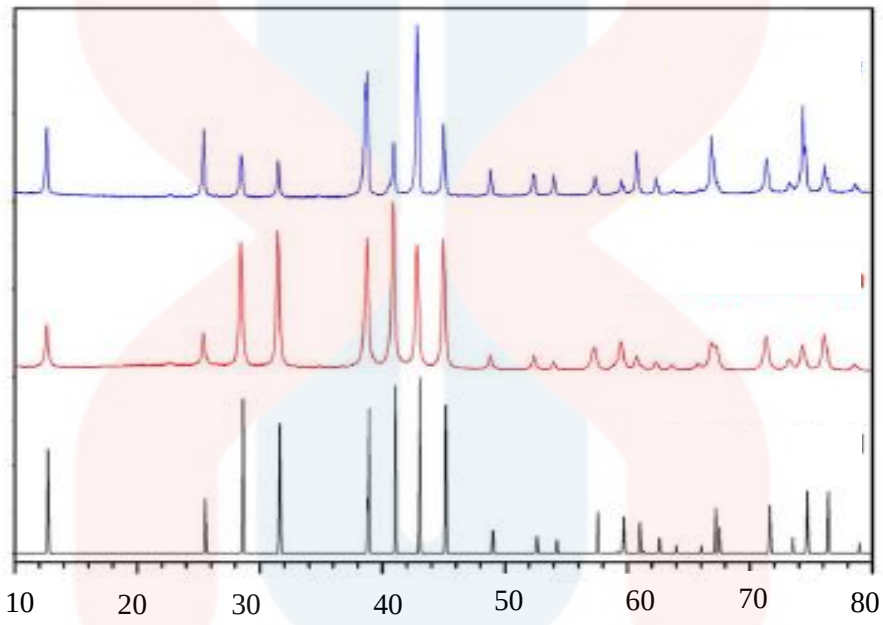

Figure 2.2 XRD pattern of alumina (Kota et al., 2016)

In periodic table of classification group elements, Al_2O_3 is form in group three and have a specification of strong interatomic bonding that result in desirable material characteristics for example high in strength and stiffness (Kumari, 2013). Besides, this material is known as very stable and robust material based on the structural design itself (Somani, 2006). The general mechanical properties of Al_2O_3 stated in Table 2.1.

Table 2.1 Mechanical properties of Al_2O_3 (Auerkari, 1996)

Mechanical Properties of Al_2O_3 (99 - 99.9 wt%)	**Value**
Flexural strength	550 MPa
Hardness	15-16 GPa
Tensile strength	310 MPa
Compressive strength	3790 MPa
Fracture toughness	4.0 MPa.m
Modulus of elasticity	330-400 GPa

Although this material called as desired characteristics, it presents some shortcomings like limitation in design due to its intrinsic stiff and brittleness. Other than that, it unable to resist deformation under load over an extended period of time according to mechanical properties on flexural strength resulted low in brittleness. Having a relatively low value of fracture toughness and resistance of thermal shock caused it least or limit in several applications, suffer in decreasing strength at high temperature as it is brittle properties (Lee, 2014; Meybody et al., 2013)

2.3 Graphite

Graphite is one of the carbon based materials and its appearance is greyish-black, opaque and has a lustrous black sheen. Carbon is widely distributed in nature and found in earth. It is also naturally crystalline allotropic forms. Graphite has good properties in mechanical, thermal and electrical. According to Gantayat et al. (2015), graphite exhibits superior properties of mechanical and electrical. Besides that, it is low density, cost and ease in processing. In addition, it also occurs as a layered material and packed closely by Van der Waals force.

According to Pierson (1993), the carbon groups include diamond, graphite, fullerene and other less common forms and all of this have same building block but different physical forms on how the buildings block was attached together and also differs in molecular or crystalline forms. Moreover, the properties between them also a bit different for example diamond is the hardest material or carbon while the other is soft compare with its. Diamond also as an electrical insulator and visible light compare with graphite which is conductor and opaque.

The schematic structure of the graphite was provided in the Figure 2.2. The structure of the graphite is built in trigonal sp^2 bonding carbon atoms and it was composed from the stacked parallel layer plane. A series of continuous hexagons deliberate as essentially infinite two dimensional molecule forms from the carbon atoms bonded to three others within the each layer plane.

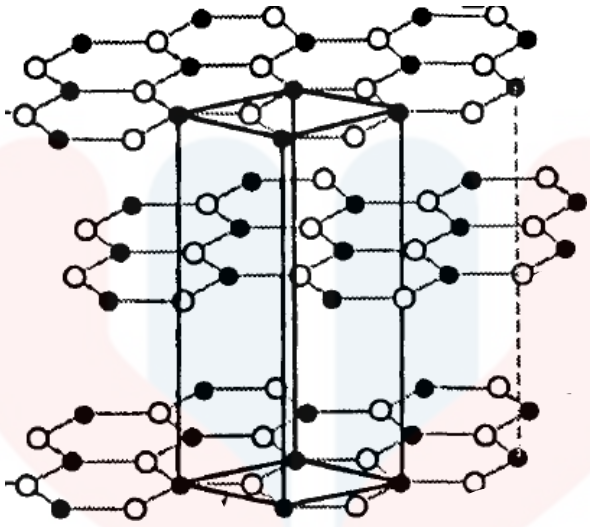

Figure 2.3 Structure of graphite (Pierson, 1993)

2.3.1 Properties of Graphite

Graphite is a carbon, a specific form of carbon. Graphite has remarkable properties such as extremely strong fiber, easily sheared lubricant, gas tight barrier and adsorbers. Physical properties of the graphite as follows hexagonal type of crystalline form, black in colour, have a lattice parameter of $a_{\circ}$= 0.264 nm, $c_{\circ}$= 0.671 nm, the estimation of boiling point at 4560 K, sublimation point at 1 atm is 4000 K, heat of fusion 46.84 kJ/mol, pauling electronegativity at 2.5 and then density at 300 K, 1 atm is 2.26g/cm³ (Pierson, 1993)

Carbon fibers usually used as reinforcement in polymer and metal matrix composite due to its properties like lightweight, high specific strength, flexibility and low thermal expansion. Besides, it is suitable for high temperature applications because of their significant creep resistance and stability (Roy et al., 2011). The XRD pattern of

graphite is shown in Figure 2.4. The pattern of the graphite displays that high peak at first and then dismiss at the end.

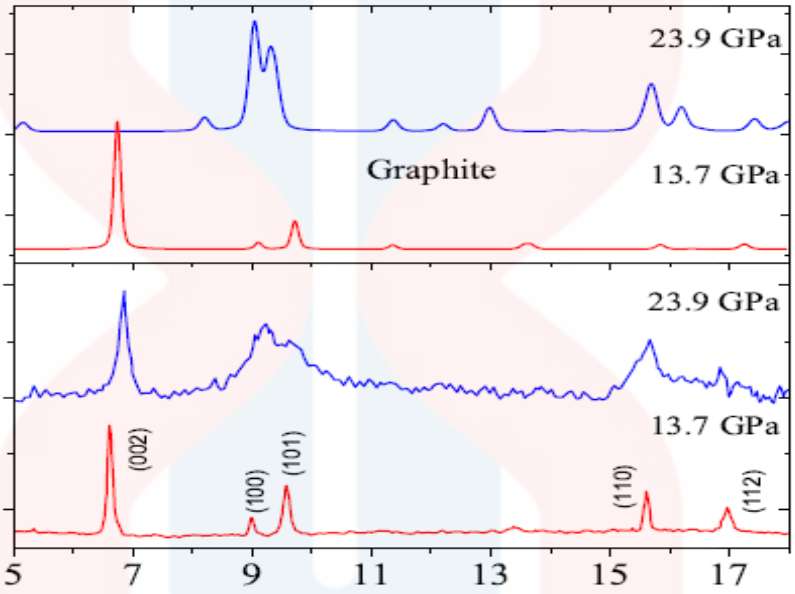

Figure 2.4 XRD pattern of Graphite (Amsler et al., 2011)

2.3.2 Mesophase Graphite

Graphite has synthetic types that were produced from various processing like vapour deposition and pyrolysis. Synthetic graphite have many categories which is carbon fibers, molded graphite and carbon, vitreous (glassy) carbon, pyrolytic graphite and carbon, carbon composites and carbon-carbon, carbon and graphite powders and particles (Pierson, 1993).

Mesophase graphite produced from the carbonization or pyrolysis process. This process is one of the direct precipitation products from the carbonaceous acetylene gas and then this process more to form a conductive graphite electrode that used in metallurgical applications such as source of energy for melting scrap iron in electric furnace, and other uses of high temperature (Tamashausky, 2006).

Figure 2.5 Mesophase structure (Adelhelm et al, 2012)

2.4 Processing of Composite

The fabrication of composite can be in various ways such as wet lay-up, prepreg lay-up, filament winding, pultrusion, press molding, resin transfer molding (RTM) or vacuum arising resin transfer molding (VARTM) and fiber placement. These all type of fabrication method gives a different quality or types of composite.

Describing the wet lay-up method is mostly said that the simple method that only need to prepared surface, cut structural plies, mixed adhesive, wet lay-up and vacuum bag. According to (Hussain et al., 2006), describe the ways of processing of composite

firstly wet lay-up which is allowed a resin to be applied only in mold, the drawbacks of this method is consisting the voids that make the product poor in mechanical properties. Next, pultrusion is one of the low cost method and high production rate in manufacturing process. RTM is a closed mold method due to its process where resin flow in the plane as well as in transverse directions of the preform (Banga et al., 2015; Hussain et al., 2006).

VARTM is the modification of the RTM that used a single sided tooling process like open molds used to make parts using vacuum. This process leads to low costs for high volume production. Filament winding is process that used wrapped over mandrel at different or same angle of a resin impregnated fibers to forms a parts.

2.5 Powder Metallurgy Processing

Processing of powder metallurgy (PM) required a several steps were powder production, powder conditioning, powder compacting or processing, sintering, sizing, and testing or inspection. All of this steps, there are three major steps in powder metallurgy is powder, processing and properties. For powder, the fabrication, size, microstructure is covered. In processing, it was include the mold, sinter, hot press and lastly properties for strength, conductivity, microstructure and density (German, 1997).

Types of PM processing conducted by ball milling process whether high energy or low energy ball milling. In PM, ball milling was acts as a problem solver to improve particle distribution and raising the well embedded reinforcing particles. Then, PM is the important technique in composite making to eliminate reinforcement isolation (Corrochano et al., 2011). Besides that, ball milling has another value like low cost,

high efficiency, low temperature synthesis, high flexibility and one of the simple process to produce wide range of fine particles materials (Chouket et al., 2016).

This method display some advantages like superior mechanical and chemical possessions according to the good chemical homogeneity, finer grain size, fabrication of complex shaped parts (Dehaghani et al., 2014). Ball milling or milling explain a collision activity occurred by the balls, and this resulted in obtaining the homogenous structure of very fine grain size (Estrada-Guel et al., 2013).

The advantage of PM technique is one of the simple and less production costs. Besides, PM is uniform distribution of reinforcing particles within the matrix and less degradation due to lower processing temperature compared with others technique. Powder metallurgy is an effective alternative to parts made by casting and forging for example, small gears and connecting rods (Corrochano et al., 2011).

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2.5.1 Milling

PM commonly involve only three routes which is milling, compaction, and sintering process. Milling process have several relevance like milling time, balls, high or low energy milling, milling design. During milling process powder particles will undergo high-energy impacts by balls. The high energy impacts result in a high amount of defects such as vacancies, dislocations, grain boundaries in particles which in turn will change the nanometer crystallite size and phase transformations (Dehaghani et al., 2014).

In milling, the cylindrical container was used to place the powders together with alumina balls to be milled. As the rods of the milling machine rotate, the container also rotates and the balls hit each other's with themselves together with the powders (Fathy et al., 2015).

The powders will be mixed homogenously for an hours. The energy required to reduce a powder to a smaller size depends on it relative changes in particle size. The time of milling depends on the particle size change, milling media size, and rotational velocity of the mill (Liu et al., 2014).

Increasing milling time, the homogeneity of particle distribution is increased and improved and will be resulted in increasing the tensile strength (Sabzevari et al., 2015). Figure 2.6 shows the illustration of how milling process occurs.

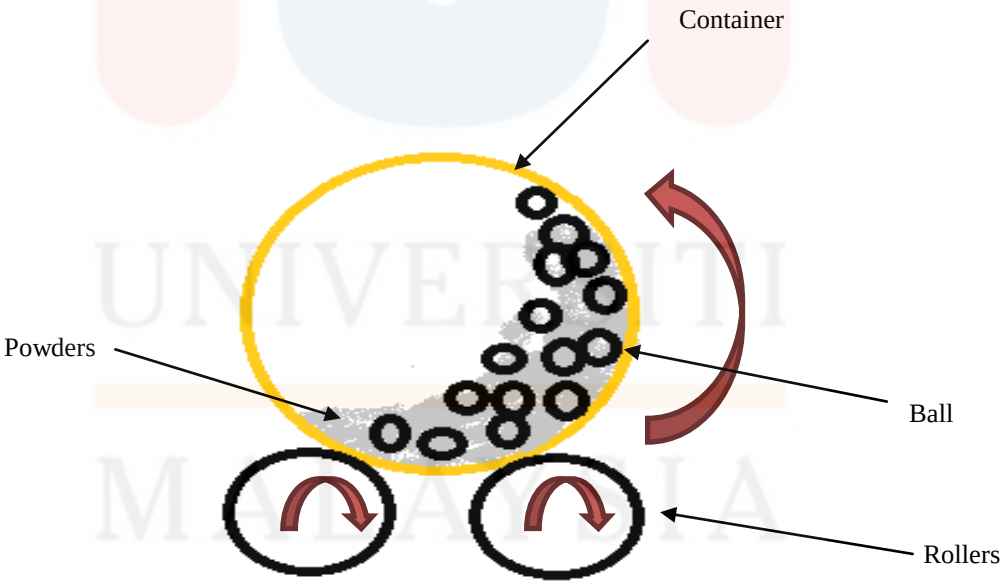

Figure 2.6 Illustration of milling process

The container rotates on the rollers that caused the alumina ball crash to each other and promote a well homogenizing between powders during milling process. Energy required enough to carry the balls to the top of the container and before falling back on the bed of material being ground (Lin and Shih, 2015; Zuhailawati and Mahani, 2009).

According to German, (1997), the energy plays an important role in milling to convey the balls rotate around the container along with the powders to be milled because if too low energy will caused the slow movement or unable to convey the balls around in the container that make the balls roll back down the container side. While, if too fast the balls move rapidly and may cause the milling process not perfect done. The conditions in the container might be one of the precautions steps that need to be covered in prevent from any unwanted factor that influenced in resulting the milling process disturb.

2.5.2 Compaction

A continuous step from the milling process, compaction process was taking place in powder metallurgy technique. Compaction is the forming process that makes loose materials powders become into desired shape with sufficient strength and specific pressure applied. According to Callister (2011) compaction also a process where the milled powders was compacted using hydraulic press machine or any machine that have a similar functions as well as hydraulic press in several amount.

The purpose of compaction was done to make a powder into preferred shape with sufficient strength to withstand from the ejection from tools and during sintering process it can be handle without any failure or breakage occurs. Compaction consists several types or procedure firstly is uniaxial, second is isostatic (or hydrostatic) and the third is hot pressing (Mahani, 2012).

These three different procedures come out with different ways of handling. Uniaxial is one of the chosen procedures because of the low in costs and high in production rate. It was simple ways which is the powders compacted in metal die by pressure that applied in single direction (Callister, 2011).

As the metallurgy final products, it was dictate that high densities. So, the reason why compaction is involved in PM is to identify or achieve powder densification. Compaction relies on external source to form powders into high density component approaches final geometry and delivering pressure caused the mechanical constraints and rate of pressurization which determine density (German, 1997).

2.5.3 Sintering

Sintering is one process that makes the bonding between the particles come together at high temperature. It is known as thermal treatment of a compaction powder at temperature below a melting point of material. During sintering, some changes occur for example shrinkage, formation of solid solution and development of final microstructure (Palmero et al., 2014; Thakur et al., 2007).

Sintering also caused surface area declines rapidly and particles sinter by atomic motion eliminates high surface energy associated with powder (German, 1997). This method contributes three stages that termed in initial stage, intermediate stage and late stage. For the initial stage, rapid growth of the interparticle neck formed and intermediate stage the structure of pore tend to smoother and has an interconnected, cylindrical nature as the compact properties developed (Callister, 2011).

Figure 2.7 describe the schematic of sintering stages occurs. The grain size growth in intermediate stage that makes grain size is larger compare with initial particle size. Then, at late stage of sintering and slow densification an isolated pores present and pores are spherical and closed with grain growth.

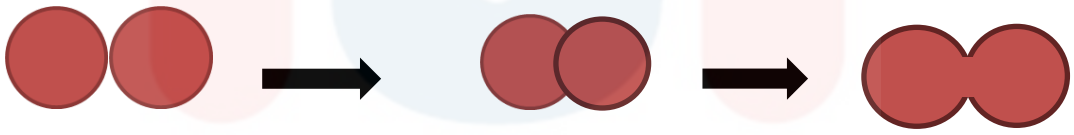

Figure 2.7 Illustration of sintering process

CHAPTER 3

METHODOLOGY

3.1 Materials

In this experiment, raw materials used are Al_2O_3 (>99.9% purity, average particle size >20 μm), and mesophase graphite (MGP) with the composition of FMGP-A-D 50~ 11μm will be used in this study to prepare Al_2O_3-MGP composite and has been purchased from Sigma Aldrich and China Steel Chemical.

3.2 Preparation of the alumina graphite

The preparation of the Al_2O_3–MGP was done using the composition of weight percentage (wt. %) using rule of mixture 70 and 30 as tabulated in Table 3.1. The overall weight in gram for these powders is 20 g, which is Al_2O_3 is 14 g and MGP is 6 g. These powders were undergoing milling process, but before running the process a solution of n-heptane was added in the powders which is 2% from the powder contain to prevent from friction occur during compaction process.

Teflon tape used to seal around the bottle cap to prevent the powders blowing out from the bottle to make sure there is no excessive powders or leakage occur in handling the powders before and after milling process done.

Table 3.1 Composition of the Al_2O_3–MGP

Material	Al_2O_3	MGP
Composition (wt.%)	70	30

Al_2O_3–MGP was undergoing milling process using low energy ball milling machine. This machine is one of the low energy balls milling machine. The real image of ball milling machine was provided in Figure 3.1. Milling time was assigned for different hours which are 15, 30, 45, and 60 h.

Before that, alumina ball was inserted into the bottle that contains these two powder mixture to increase the effectiveness of the milling process. The weight of alumina ball used in this experiment was 200 g. This ball cleaned before insert into the PE bottle with sand and milling for 1 h, and then washed with water flow to eliminated impurity from the alumina ball.

This step was repeated for each new powders and different milling time. The schematic illustration for the set up process of milling is shows in Figure 3.2. The alumina ball was inserted with the powder ratio of 10:1 and this ratio is persistent for overall in experimental work. The speed of rotational for ball milling was set to 100 rpm.

Figure 3.1 Real image for low energy ball milling machine

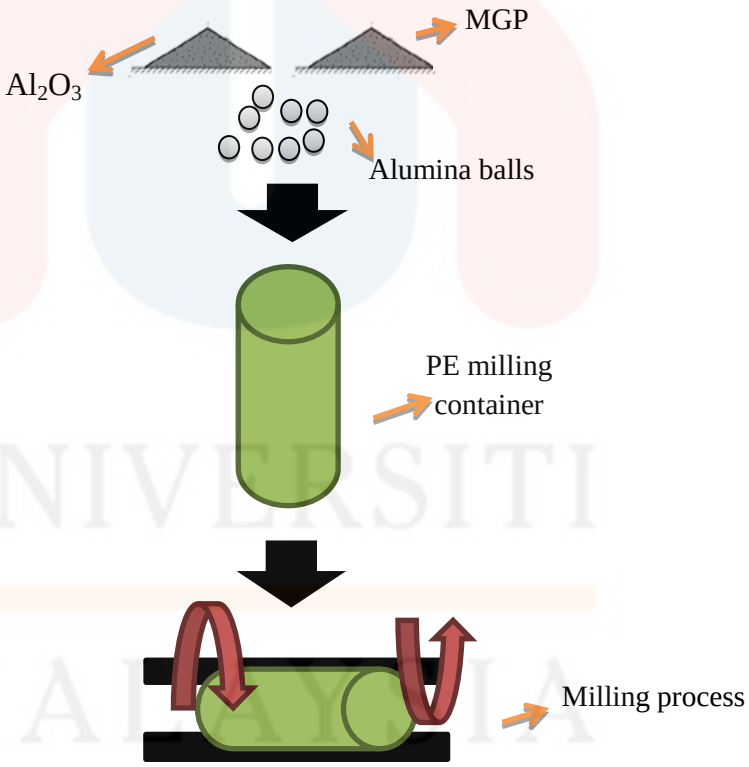

Figure 3.2 Illustration of milling experimental setup

3.3 Powder compaction

After the milling process was done, Al_2O_3–MGP powder was compacted using Specac manual hydraulic press machine with stainless steel die with diameter of 10 mm as shown in Figure 3.3 at room temperature by cold die compaction method. The mold was cleaned with lubricant before and after the powder was poured for every each of pallets making. In compaction, the powder was mixed with 1 % of zinc stearate to avoid the friction along the die wall and the pressure of the compaction was done with different pressure which is 200, 400, 600, and 800 MPa.

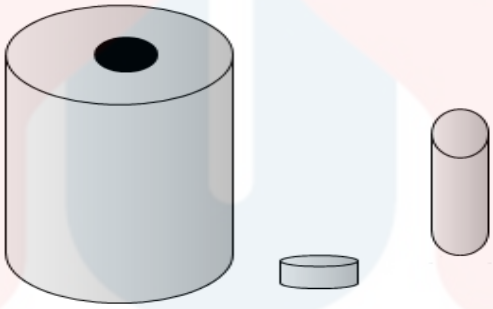

Figure 3.3 Schematic image of Stainless steel die mold

The different pressure used is purposely to investigate the effect of compaction pressure on the milling powders. During the compaction for each pressure, it needs to hold on for two minutes to prevent from the agglomeration of fine powder during take out from the die. Before poured the powder into the mold, it was weighed for 1.1-1.5 g for each pellets of composite.

Figure 3.4 shows the real apparent of the hand press machine used to compact the Al_2O_3–MGP powder to become a pallets. The compacting was done in uniaxial single action manual hydraulic press. The process of pressure compact occurs to produce powder pallet was assembled in Figure 3.5. The pallet was dismantle after the compaction done.

Figure 3.4 Real image of hydraulic press machine

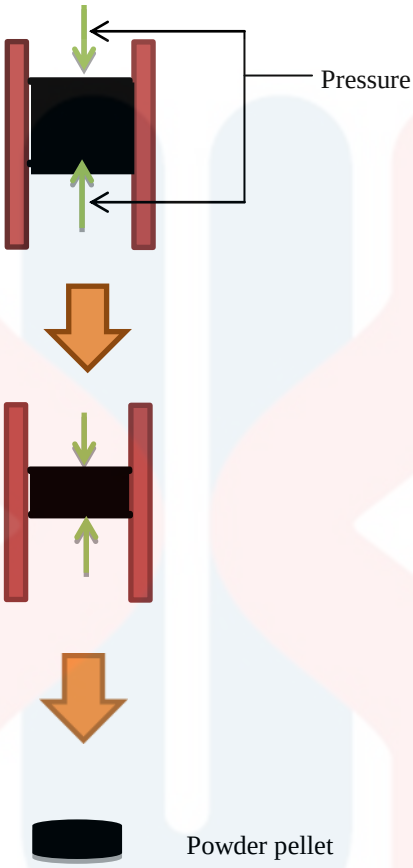

Figure 3.5 Pressure of compaction powder pallet

3.4 Characterization of Alumina Graphite Composite

For the characterizations of Al_2O_3–MGP composite, the analytical methods used to analyse the results or characteristics of the composite sample are x-ray diffraction (XRD), optical microscope (OM).

3.4.1 X-ray Diffraction

The phase identification of Al_2O_3–MGP composite was analysing using XRD analyser (D2 phases, Bruker). The Cu Ka radiation (λ = 0.154mm) will be used in the scan range of 20^{o} to 80^{o} of 2θ angle with step size of 0.02^{o}. The Software DIFFRAC. EVA phase identification of X-Ray diffraction pattern will be used to perform qualitative and quantities analysis of the Al_2O_3–MGP composite. Crystallize size, internal strain, dislocation density and lattice parameter will be reveal using qualitative analysis. Besides, it also can give the percentage of phase present in Al_2O_3–MGP powder.

WH is the most common method used to evaluate the crystallite size and internal strain in powders. The assumption is that the whole line broadening, B_o is a sum of the total broadening of size, lattice strain and instrument (Cullity and Stock, 2001) as in equal:

$$B_o = B_i - B_{crys} + B_{strain} \quad \text{(Eq.1)}$$

Where B_i broadening due to instrumental, B_{cryst} broadening due to size and Bs_{train} broadening due to strain. Subtracting the instrumental effect, equation 2 (Cullity and Stock, 2001) becomes:

$$B_r = B_{crys} + B_{strain} \quad \text{(Eq.2)}$$

Therefore, due to crystallite size and internal strain, WH method is given by (Williamson and Hall, 1953) as equation 3:

$$B_r \cos\theta = \frac{k\lambda}{D} + \eta \sin\theta \qquad \text{(Eq.3)}$$

3.4.2 Optical Microscope

The morphology of the Al_2O_3–MGP powders with different milling time was analysed using optical microscope (OM) to see the differences of their microstructure. This OM is type of microscope that used a visible light and systems of lenses for magnifying the images of small samples. The Al_2O_3 –MGP powders was put on the slide with very thin layer and placed under the microscope to see their morphology. The different magnification used in this analysing to get the optimizing and clear images.

The particles size of the powder will be observed where to identify there are some effect or not on the different milling times used. The distribution of the two different powders will be examined through the microstructure of raw materials and mixed powder.

3.4.3 Density

A density is need in level of performance. The high density of the material will perform high in performance. The dimension and mass of composite will be measured after compaction process was done . Then, green density (GD) of the composites will be calculated using general theory equation after measure the dimension and mass of composite pellet compation. In order to determine the GD of composite:

$$GD = \frac{m}{V} \quad \text{(Eq. 4)}$$

where: m is a mass, v is a volume.

In order to study the compressibility of produced composite, densification parameter should be calculated. Densification parameter will be determined using equation 5

$$\text{Density} = \frac{GD-AD}{TD-AD} \quad \text{(Eq. 5)}$$

where: GD is a green density, AD is for apparent density and TD is theoretical density.

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3.5 Overall Research Flow Chart

The overall procedure for the experimental for preparations of alumina graphite composite was summarized in Figure 3.6.

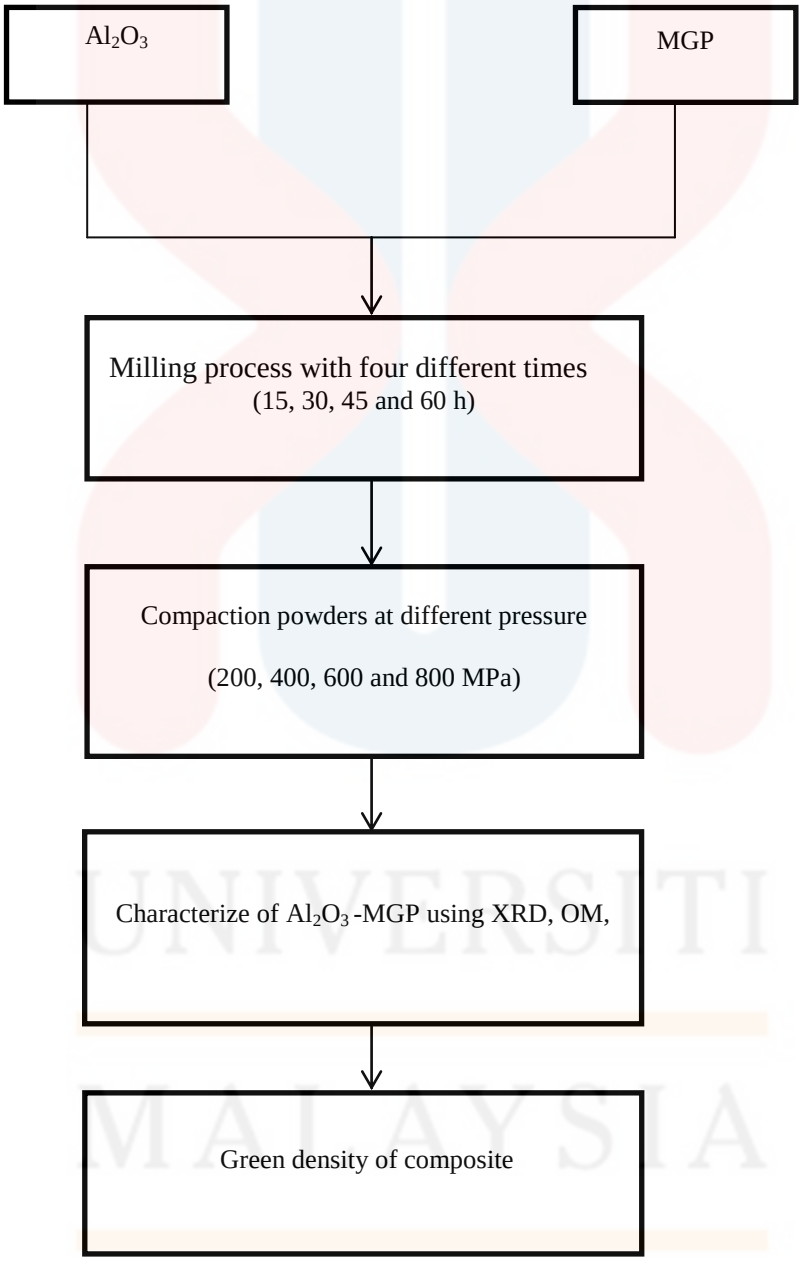

Figure 3.6 Flowchart of experimental

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

In this section, the results from the experimental of this study were discussed based on the types of analysis that have been done. The result that was discussed is X-ray diffraction (XRD) pattern phase identification of raw materials and milled powders. The effect of milled powders in crystallite size, green density, densification parameter and microstructure was observed. The effect of compaction with different pressure applied according each milling time also was deliberated.

4.2 Characterization of Raw Materials

XRD was used to analysis the characterizations phase identification of the raw materials of Al_2O_3 and graphite. The results obtained from XRD for raw material Al_2O_3 and graphite as shown in Figure 4.1. Peaks form between the Al_2O_3 and graphite shows the comparisons as we can see clearly in Figure 4.1. These powders were examined that have crystalline structure.

For the Al_2O_3 the best three peaks that we get from the XRD analysis is at position of 2θ at 35.137º, 43.341º and 57.483º and peaks position of hkl at $(\bar{1}\,1\,4)$, $(\bar{2}\,1\,\bar{3})$ and $(\bar{2}\,1\,\bar{6})$ respectively and other peaks indicate the broadening and short as well as in the previous research for this material pattern in XRD (Kota et al., 2016). The present numbers of peaks in Al_2O_3 shows that it is strong crystalinity and corundum phase.

The disappearance peaks in phase identification of XRD meaning that dissolution occurs of second element into the matrix and form a solid solution. Then the three strong peaks 26.538º, 44.520º and 83.527º belongs to graphite at hkl of (0 0 $\bar{2}$), ($\bar{1}$ 0 $\bar{1}$) and ($\bar{2}$ 1 $\bar{2}$). From the observation, graphite designate as a crystallinity based on the results that present of peaks.

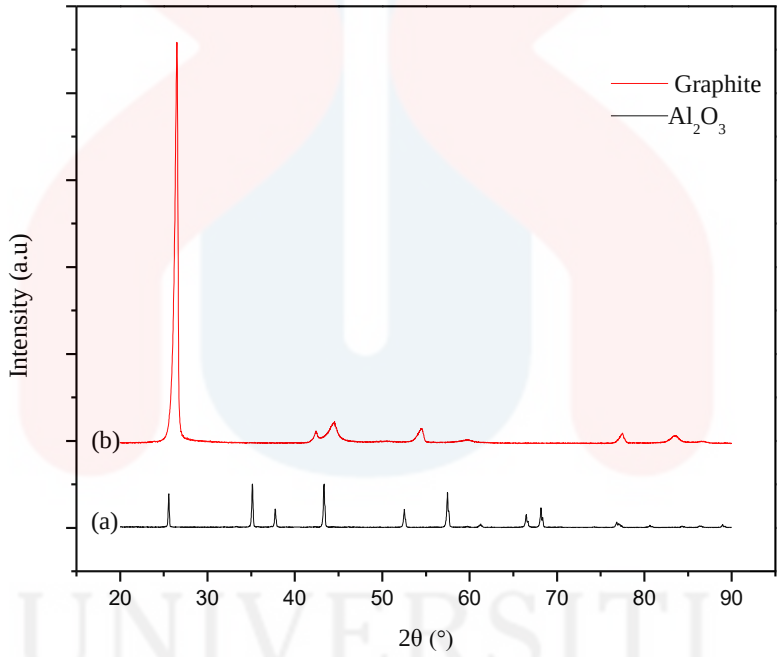

Figure 4.1 XRD patterns of (a) Al_2O_3 and (b) graphite

4.3 Effect of Milling Time

The purpose of this experimental was done to assess the effect of the structural and microstructural of milled powders at different milling time which was conducted at 15, 30, 45, and 60 h. Others parameter was persistent including the speed of the milling, balls size, N-heptane solution, and then the types of container used on low energy types of ball milling machine.

4.3.1 X-ray Diffraction

The XRD pattern of the milled powders was shown in Figure 4.2. The pattern shows milled powders with different time of milling (h). During milling process was carried out n-heptane solution was added to prevent from the agglomeration of the mixed powders during milling takes place.

As we can see, the milled powders show the different with the pattern of raw materials of XRD. The milling time at 15 h, the peaks seem too overlapped because the peaks between Al_2O_3 and graphite at 2θ of 43.37º and 43.45º respectively look much closed with each other's and this due to the reaction takes place which is graphite powders diffuse into the Al_2O_3 powders cause particle powder reduce in size of particle powders and make the peaks become shorten.

At 30 h the XRD pattern of milled powders shows there is no overlapped occurred between these two different powders. Its same goes with another three different milling times which are 45 and 60 h. The XRD pattern of all this parameter in experimental give the results that these powder more to crystalline structure.

All of the results give the expression that there is no unfamiliar or any new realization peaks or phase. A little variation that has been noticed in this situation is at high in milling time (h) done, the peaks of graphite and Al_2O_3 were decrease. This happen because increasing the milling time, the powders become decreases in size and more homogenous.

The peaks of graphite diminished due to solid solution behavior of alumina that make graphite diffuse into alumina graphite. Besides, after milling process was done the particle powders undergo reduce in size that make the peaks disappear or wider. As the increasing milling time, the grain size will be refining as the homogenously milled.

Although the peaks of the milled powders become wider and seems close to each other but the pattern still shows the different elements present in powder milled of XRD analysis according to the peaks of crystallinity of the elements from the raw materials as a reference.

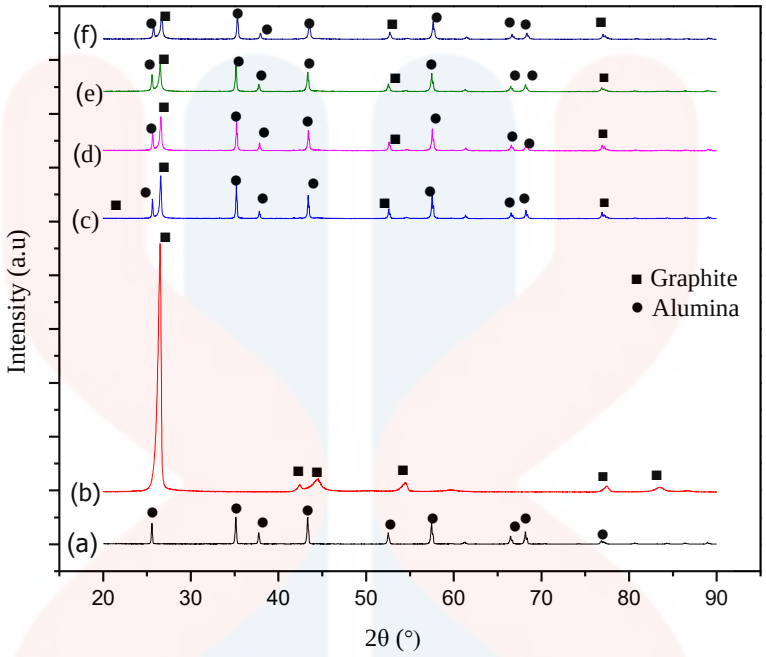

Figure 4.2 XRD patterns of raw materials (a) Al_2O_3 (b) Graphite and time of milled powders (c) 15h (d) 30h (e) 45h and (f) 60h with alumina balls.

4.3.2 Crystallite size and internal strain of milled powders

The plot of the Br Cos θ against sin θ actually indicate to the calculating of the crystallite size and internal strain of the milled powders that gives the results always increasing in each milling time (h) but with the different value.

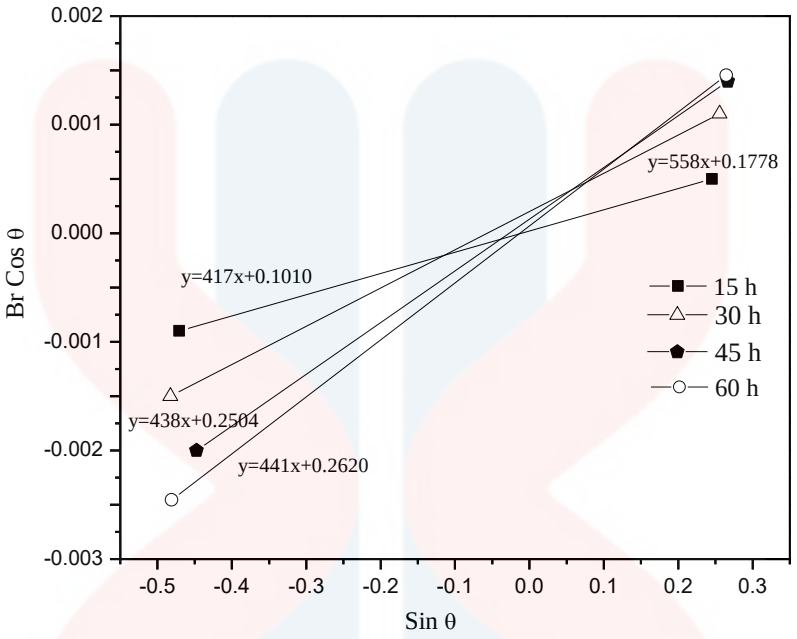

Figure 4.3 Plot Br Cos θ against sin θ for calculating crystallite size and internal strain of milled powders

Br Cos θ against sin θ will form a intercept that resulting in the internal strain of powder milled and the the gradient from the equation of this will give the value of crystallite size. This two relations come out with the low crystallite size will cause high in internal strain with high milling time. Based on Figure 4.3, the best milled powders of this intercept is at 45 h time of milling. At 45 h, the crystallite size drop from 558 to 438 nm.

Crystallite size of the milled powders shows the results from the cube root of the volume as same size and shape. By increasing the time of milling, the size of crystalline should decrease as the high in rate over time because of the repeating deformation occurs (Sivakumar et al., 2004). The graph in Figure 4.4 shows between 15 and 30 h of milling time, crystallite size shows the increasing reading from 417 to 558 nm. This might happen at 30 h of milling time because of the milling process failure due to the low energy collisions between balls to break the particle powders.

Besides, at 30 h of milling time, as crystallite size is high and this might happen during milling process because of the agglomeration occurs within the powders and caused defect dislocation or vacancies that make milled powders not homogenizing properly. Moreover, another reasons is come from the energy supply from the machine is slow which make balls rolls back to the ground of the milling container (R.German, 1997). However, at 45 h the reading of crystallite size is display the reduction of 438 from 441 nm and then at 60 h demonstrate only a slight increase.

For milling time at 15h, the crystallite size was control from the value of full width of half maximum (FWHM) at 25.629 and 57.529 of peaks ($\bar{1}$ 1 $\bar{2}$), and ($\bar{2}$ 1 6), respectively. For 30h, the peaks selected at ($\bar{1}$ 1 $\bar{2}$) and ($\bar{2}$ 1 6), at 45h the strong peaks were ($\bar{1}$ 1 $\bar{2}$) and ($\bar{2}$ 1 $\bar{6}$). The highest milling time which is 60 h, the peaks is ($\bar{1}$ 1 $\bar{2}$) and ($\bar{2}$ 1 $\bar{6}$). The reasons of the theoritically of crystalline size said that increase milling time resulted dropped crystallite size because of the starter of defects form like dislocations within grain during milling process takes place.

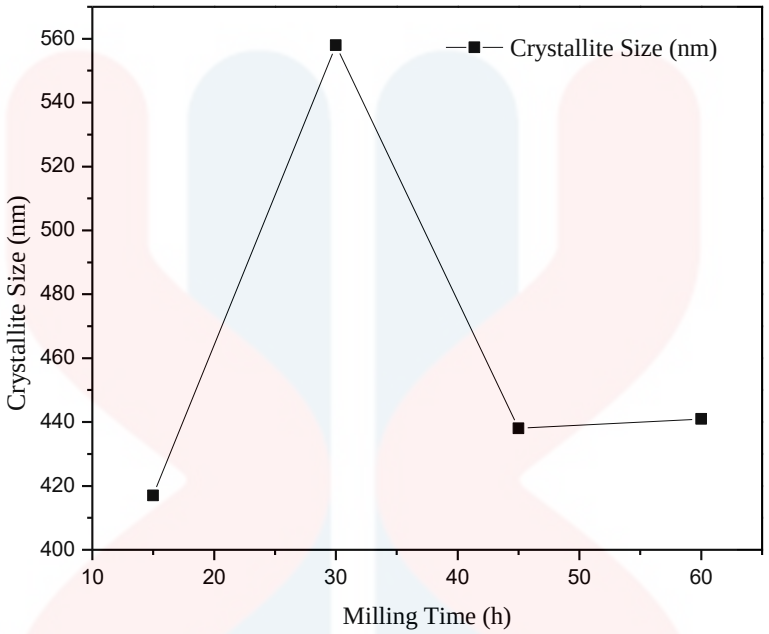

Figure 4.4 Crystallite size against the different of milling time

Internal strain of the milled powders caused from the energy that supplied for the milling process occurs and resulted the formation of the internal stress for the composite. Consequence from the different time of the milling time, the internal strain also indicate the different results.

Strain was called as a when external forces are applied to objects and give the changes in size and shape of the objects or easilly understood by term of strain is a relative change in shape or size of an object due to externally applied forces. In this experiment, the external forces was indicate to the energy supply during the milling process.

In crystal, strain was classified with two types which is called that uniform and non-uniform strain. Uniform strain defined that a changes in a unit cell parameter peak shiftening take place. This caused the unit cell expand and contract in isotropic ways and then this type of strain resulted there is no broadening linked.

While, non-uniform strain have the different process with uniform strain due to its changes in their atoms that systematicaly shifts from its ideal positions and to peak broadening. Point defects like vacancies and site disorder, plastic deformations or poor crystallinity signpost to the rise of this type of strain.

In this experiment, the parameter selected was the different of milling time for each composite powders to analysis or to prove the theory above state that increasing the milling time, the increasing internal strain. Others things are keep constant for example amount of powders, weight of balls, and energy applied.

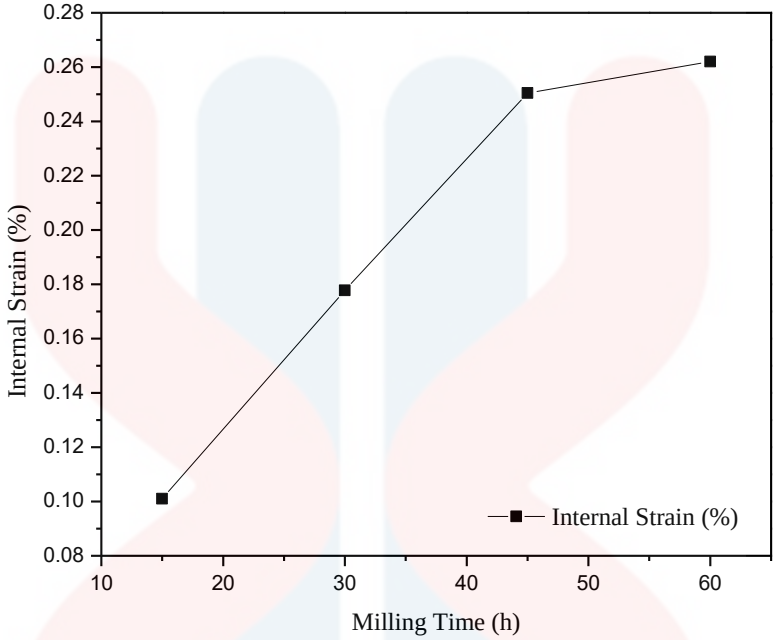

Figure 4.5 Internal strain of different milling time

Figure 4.5 display the graph of internal strain (%) against milling time (h) of the milled powders, pattern of graph can be conclude that by increasing the milling time (h), the highest the internal strain (%) occurs. At 15 h milling time, the internal strain shows the least in value. The increasing of internal strain occurs at 30 and 45h respectively due to stress produced because of the collisions between the balls and the powders are long in time compared to 15 h. William son (WH) method was used to identify this internal strain based on the milling time. Homogenous powders milled produced finer powders that reduce in crystallite size and resulted high internal strain.

Other than that, the phenomena that increasing the internal strain can be state that because of the strain hardening of the powders due to the ball milling. During 60 h time of milling, the graph did not show the rapid increasing of internal strain like before at 30 and 45 h. This might because of the internal strain reach the optimum value in milling process. Figure 4.6 shows the different in increasing of internal strain and the status of the crystallite size that various in increasing and decreasing at some different milling time (h).

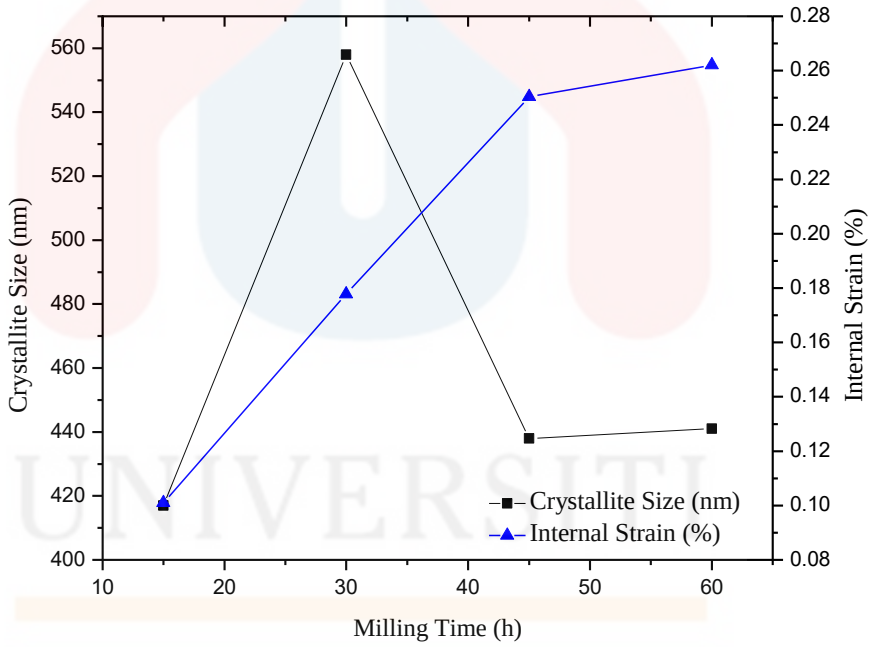

Figure 4.6 Crystallite size and internal strain of milled powders

4.3.3 Microstructure of Raw Materials

The microstructure of raw materials or pure Al_2O_3 and graphite before mixed together for milling process was observed under optical microscope (OM) as provided in Figure 4.7. Before milling process was done, n-heptane solution was added in the container to prevent agglomeration occurs. As we can see the microstructure of Al_2O_3 at (a), the particle is bigger compare with the particle size of pure graphite at (b) which is quite small than Al_2O_3.

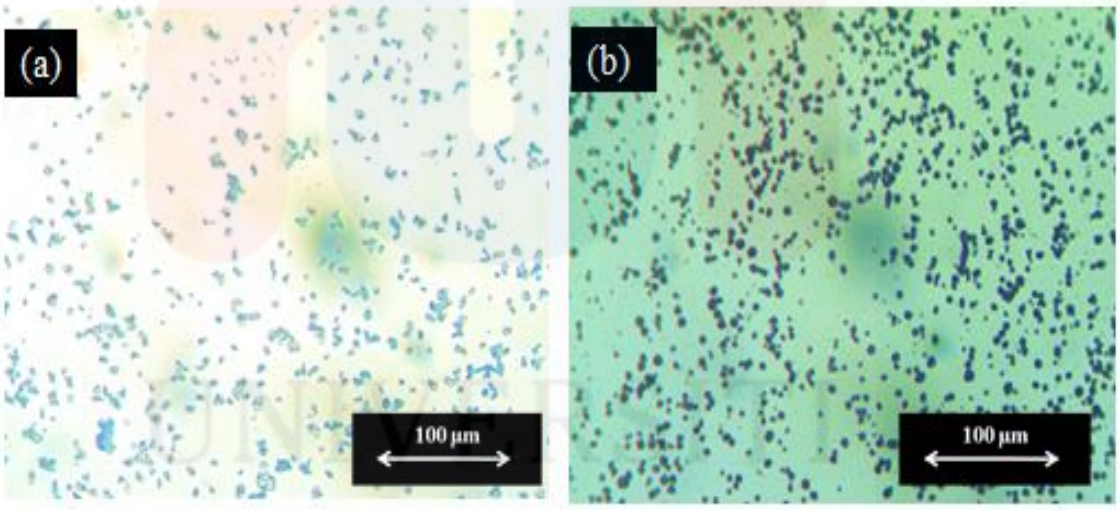

Figure 4.7 Microstructure of raw material

4.3.4 Microstructure of Milled Powders

The microstructure of the milled powder for 15 h (a) in Figure 4.8 display that the size of particle is being mixed together and seems like a little agglomeration present. This is because the powders do not being milled homogenously yet. The distributions of particle size also different between all the different periods of milling time (h). The shape and particle size of the microstructure also changing.

At 60 h milling time, the microstructure illustrates the smaller size of particle and the distribution of particle is uniformly. The agglomeration detect free at this hours due to its homogenously powders was milled by adding the n-heptane to prevent the agglomeration present.

The fine grain or microstructure produces at 60 h. The long period time of milling, the finer and smaller microstructure will harvest. The fracture break occurs which is the fraction from the bigger size to smaller. Changing the microstructure from coarse to fine grain size. Figure 4.8 indicate the effect of microstructure with different milling time.

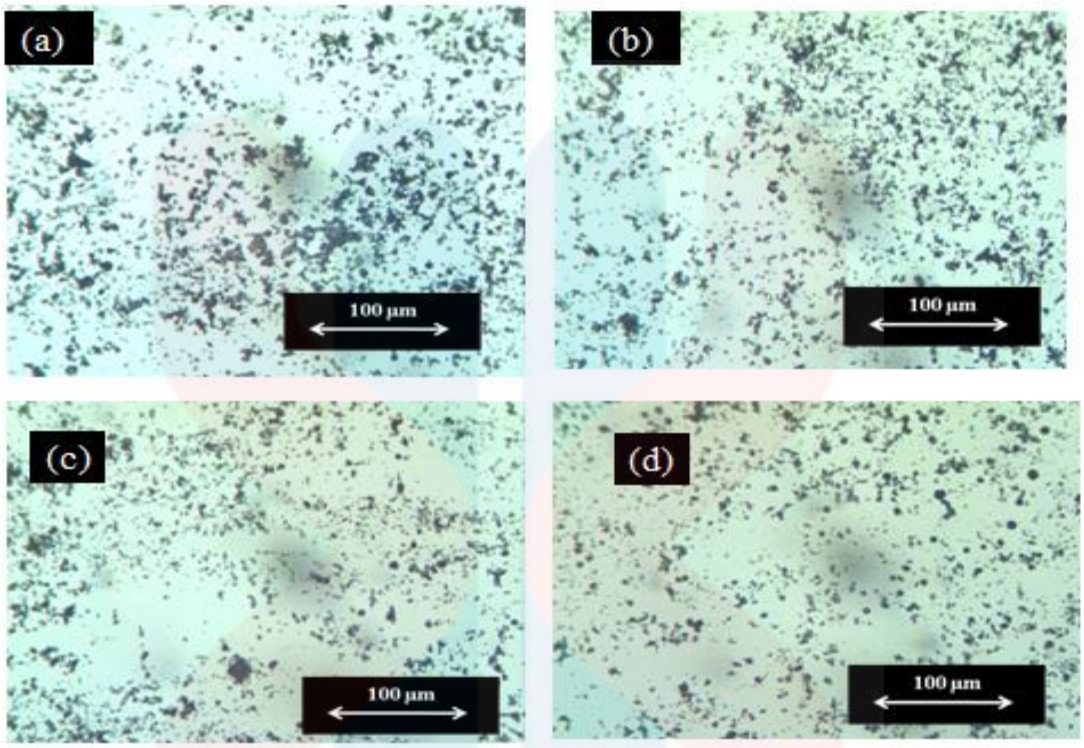

Figure 4.8 Microstructure of milled powders

4.4 Properties of milled powders

The properties of this composite were conducted through the density testing or analysis after compaction of the powders was done. The different compaction pressure was done to each of milling time (h).

4.4.1 Green Density

Different compaction pressure was done to calculate the density of the powders. The pressures involved are at 200, 400, 600, and 800 MPa for each different milling time (h). Compaction also was done to increase the density of the materials. The purpose of the compaction is to reduce the porosity of the material and to minimum the voids in the materials itself. High pressure cause low volume porosity due to the particle fracture occurs and the graphite fills voids in Al_2O_3.

Hydraulic press machine was used to compact the powders in axial loading with 10 mm stainless steel mold. Figure 4.9 shows the green density (%) with different pressure of compaction express the increasing of the compaction pressure resulted in increasing the green density of the powders.

Arising the compaction pressure reduce the volume of the powders. Compaction pressure high will resulting the high density, this make the particle size fill the voids that form in the powders which help to rid the flaws. At 45 h time of milling shows the high in density and as an optimum time for green density for compaction. During the compaction, the powders that easily to compact brings the brittle behavior and for difficult it means that the powders is ductile.

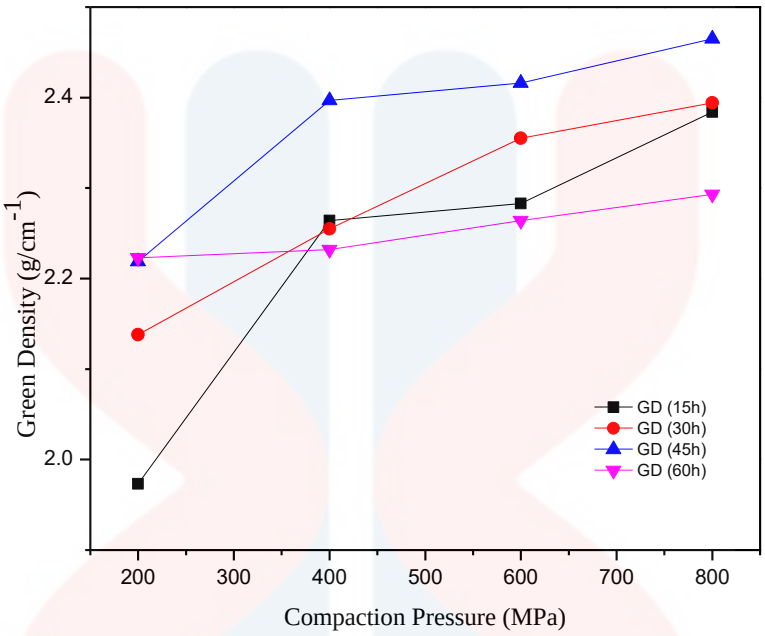

Figure 4.9 Green density (g/cm-^{1}) of the compaction pressure (MPa) at different milling time (h)

4.4.2 Densification Parameter

Densification parameter as shown in Figure 4.10 demonstrates that the pattern of graph is likely with the green density graph. This is because they are related with the value of green density. The relationship of the green density and densification parameter is proportional. Composite become denser when increasing compaction pressure.

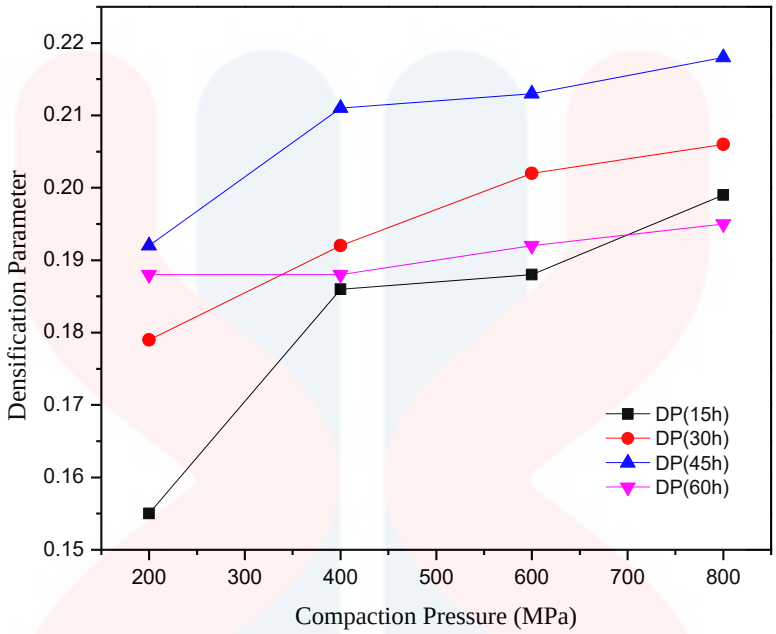

Figure 4.10 Graph of densification parameter against compaction pressure

Another graph was plotted to recount the compaction pressure with the density in Figure 4.11. The concept of the graph was proposed by the founder Panelli and Ambrozio Filho (Panelli and Ambrozio Filho, 1998) with this equation:

$$\ln\left[\frac{1}{1-D}\right] = AP^{\frac{1}{2}} + B \qquad \text{(Eq. 4.1)}$$

where: D is a relative density, and P is a compaction pressure.

The relationship between this two letter give the result in *A* that refer to the value calculated from the equation above. According to Panelli and Ambrozio-Filho (1998), the value of *A* represent different type of materials used which is when high value *A* indicate to soft powders used whereas low *A* shows the ceramic materials. It can be stated that parameter A of the compressibility curve provides the plastic deformation capacity of the powder in compaction.

The graph in Figure 4.11 shows the relation between density with plastic deformation. Pressure applied to contact area between grains increase tendency to particle powder become plastic deformation. At 45 h, high plastic deformation enhance by high density of powder.

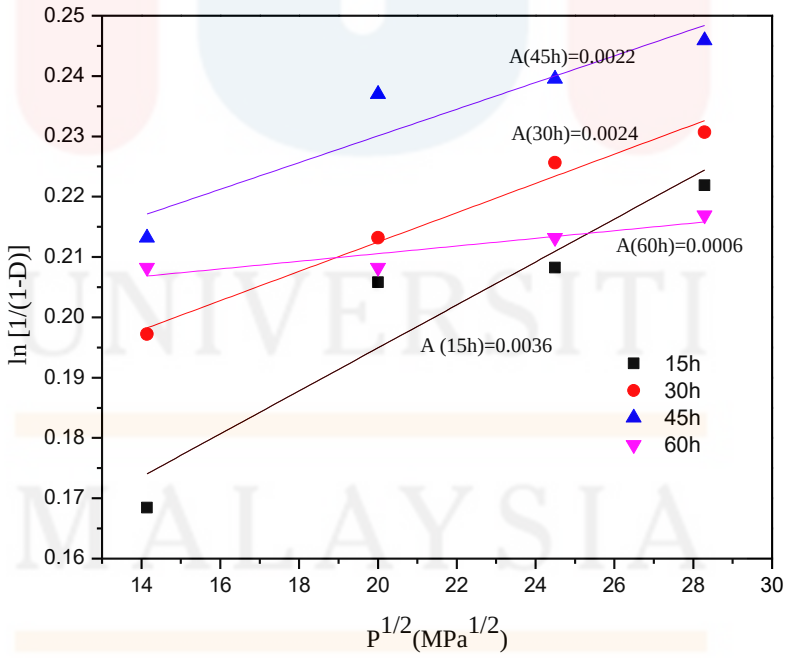

Figure 4.11 Plot of experiment data for green compact using the equation proposed by Panelli and Ambrozio Filho (1998)

CHAPTER 5

CONCLUSION

5.1 Conclusion

Based on the experimental results, the conclusion that can be achieved is when the different milling time used in the experiment, the different effect of the powders will form. It can be conclude that the dissimilarity of the milling time affect the microstructure and structural of the powders. Same goes with the deviation in XRD identification of the raw materials with the milled powders shows the variations. Finer microstructure will achieve in increasing period of milling time.

Al_2O_3-MGP composite was positively done and the bulk of composite done by compaction step. Compactions pressure with different amount will give a different affect. Higher compaction pressure applied on the powders will decrease the volume and voids of powders. The internal strain is directly proportional with the milling time, as higher milling times give high internal strain.

Increasing milling time applied give the effect for microstructure from coarse to finer particle size and phase peaks become shorten in increasing time. For the high compaction pressure, increase green density, densification parameter and plastic deformation of alumina graphite composite. Last but not least, the objectives of the study Al_2O_3 graphite composite by powder metallurgy was successful achieve.

5.2 Future Work Suggestions

In powder metallurgy technique, there are some parameters that can be done to study the effect of powders during milling time. In future study, the high energy milling machine was suggested used to see the differences between high energy and low energy. Besides that, in milling the variations size of balls can be used as a parameter.

During milling, the uses of gas in the container or jar might help for a better result because the milling is do in vacuum condition which means that there is no unwanted or any factors that disturb the milling process for example oxygen gas in container.

Sintering process should be done to carry out the testing for the composite. Mechanical testing can be done to investigate the strength of the composite materials after sintering takes place. The alteration of the compaction pressure also can give the different results in density of composite.

The types of milling machine used, in further study high energy milling machine can be used to compare or to observe the differences that might occurs rather than low energy milling machine. This can be investigating according to energy supply for the balls to mill in the container for producing high homogenous powders.

To improve the investigation in this field, the use of optical microscope should be improved by using scanning electron microscope (SEM) or another advance of microscope which is transmission electron microscope (TEM) for a better result of microstructure. The 3D images will contribute sharp images that can see the changes of microstructure clearly.

Surface analyser using brunauer-emmett-teller (BET) for surface area of solid material can be carried out in this further study. Specific surface area of composite can be measured using this machine. This machine refers to adopt non- corrosive gases like nitrogen, argon, carbon dioxide for determination of the surface area data through multi-layer adsorption.

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REFERENCES

Adelhelm, P., Cabrera, K., & Smarsly, B. M. (2012). On the use of mesophase pitch for the preparation of hierarchical porous carbon monoliths by nanocasting, *6996*(November 2016).

Aigbodion, V. S., Agunsoye, J. O., Kalu, V., Asuke, F., Ola, S., Metallurgical, N., & Centre, D. (2010). Microstructure and Mechanical Properties of Ceramic Composites. *Journal of Minerals & Materials Characterization & Engineering*, *9*(6), 527–538.

Amsler, M., Flores-livas, A., Lehtovaara, L., Balima, F., Alireza, S., Willand, A., … Marques, M. A. L. (2011). The crystal structure of cold compressed graphite, 1–4.

Auerkari, P. (1996). Mechanical and physical properties of engineering alumina ceramics.

Banga, H., Singh, V. K., & Choudhary, S. K. (2015). Fabrication and Study of Mechanical Properties of Bamboo Fibre Reinforced Bio-Composites, *6*(1), 84–99.

Baptista, R., Mendão, A., Rodrigues, F., & Guedes, M. (2016). Effect of high graphite filler contents on the mechanical and tribological failure behavior of epoxy matrix composites. *Theoretical and Applied Fracture Mechanics*.

Callister, W. D. (2011). *Materials Science* (8th ed.). John Wiley & Sons.

Cho, J., Chen, J. Y., & Daniel, I. M. (2007). Mechanical enhancement of carbon fiber / epoxy composites by graphite nanoplatelet reinforcement, *56*, 685–688.

Chouket, A., Cheikhrouhou, A., Optasanu, V., Bidault, O., & Khitouni, M. (2016). AC SC. *Journal of Alloys and Compounds*.

Clau, B. (2008). Fibers for Ceramic Matrix Composites, 1–20.

Corrochano, J., Lieblich, M., & Ibáñez, J. (2011). The effect of ball milling on the microstructure of powder metallurgy aluminium matrix composites reinforced with MoSi2 intermetallic particles. *Composites Part A: Applied Science and Manufacturing*, *42*(9), 1093–1099.

Dae-Yeop Lee, D.-H. Y. (2014). Properties of alumina matrix composite reinforced with SiC whisker and carbon nanotube. *Ceramics International*, 1–26.ceramint.2014.06.030

Dehaghani, M. T., Ahmadian, M., & Fathi, M. (2014). Effect of ball milling on the physical and mechanical properties of the nanostructured Co – Cr – Mo powders. *Advanced Powder Technology*.

Emad Omrani, Afsaneh Dorri Moghadam, Mahmoud Algazzar, Pradeep L.Menezes, P.

K. R. (2016). Effect of graphite particles on improving tribological properties Al-16Si-5Ni-5Graphite self-lubricating composite under fully flooded and starved lubrication conditions for transp ... Effect of graphite particles on improving tribological properties Al-1, (September).

Fang, T., Hsiao, L., Duh, J., & Sheen, S. (2006). A novel composite negative electrode consist of multiphase Sn compounds and mesophase graphite powders for lithium ion batteries, *160*, 536–541.2006.01.034

Fathy, A., El-Kady, O., & Mohammed, M. M. M. (2015). Effect of iron addition on microstructure, mechanical and magnetic properties of Al-matrix composite produced by powder metallurgy route. *Transactions of Nonferrous Metals Society of China (English Edition)*, *25*(1), 46–53.

Galusek, D., & Galusková, D. (2015). Alumina Matrix Composites with Non-Oxide Nanoparticle Addition and Enhanced Functionalities, 115–143.

Gantayat, S., Prusty, G., Rout, D. R., & Swain, S. K. (2015). Expanded graphite as a filler for epoxy matrix composites to improve their thermal , mechanical and electrical properties. *New Carbon Materials*, *30*(5), 432–437.

Geric, K. (n.d.). Ceramics Tool Materials , *3*, 367–372.

Hussain, F., Hojjati, M., Okamoto, M., & Gorga, R. E. (2006). Journal of Composite Materials.

Hussainova, I., Baronins, J., & Drozdova, M. (2016). Author ' s Accepted Manuscript. *Wear*.

J.Lamon. (2011). Ceramic reinforcements for composites. *National Institute of Applied Science*, *3*, 51–85.

Journal of Alloys and Compound s Effect of process parameters on micro and macro-properties of an Al-based nanocomposite prepared by means of mechanical milling. (2013). *Journal of Alloys and Compounds*, 2–6.

Kota, S., Zapata-solvas, E., Ly, A., Lu, J., Elkassabany, O., Huon, A., … Barsoum, M. W. (2016). Synthesis and Characterization of an Alumina Forming Nanolaminated Boride : MoAlB. *Nature Publishing Group*, (April), 1–9.

Kumari, S. (2013). Phase Evolution , Densification , Microstructure And Mechanical Properties Effect of TiO 2 Addition in Al 2 O 3 : Phase Evolution , Densification , Microstructure And Mechanical Properties, (109).

Li, W., Zhang, C., Liao, H., Li, J., & Coddet, C. (2008). Surface & Coatings Technology Characterizations of cold-sprayed Nickel – Alumina composite coating with relatively large Nickel-coated Alumina powder, *202*, 4855–4860.

Lin, H. Y., & Shih, C. Y. (2015). Efficient one-pot microwave-assisted hydrothermal synthesis of M (M = Cr, Ni, Cu, Nb) and nitrogen co-doped TiO2 for hydrogen

production by photocatalytic water splitting. *Journal of Molecular Catalysis A: Chemical*, *411*, 128–137.

Liu, J., Lin, L., Li, J., Liu, J., Yuan, Y., Ivanov, M., & Chen, M. (2014). Effects of ball milling time on microstructure evolution and optical transparency of Nd : YAG ceramics. *Ceramics International*, *40*(7), 9841–9851.

Mahani Yusoff. (2012). *Processing, Microstructure and Properties of In Situ Copper Reinforced Tungsten Carbide Nanostructured Composite*.

Meybodi, S. M., Barzegar Bafrooei, H., Ebadzadeh, T., & Tazike, M. (2013). Microstructure and mechanical properties of Al2O3–20wt%Al2TiO5 composite prepared from alumina and titania nanopowders. *Ceramics International*, *39*(2), 977–982.

Mishra, S. K., Gokuul, V., & Paswan, S. (2014). Int . Journal of Refractory Metals and Hard Materials Alumina-titanium diboride in situ composite by self-propagating high-temperature synthesis (SHS) dynamic compaction : Effect of compaction pressure during synthesis. , *43*, 19–24.

Neil N. Gow, G. P. L. (1993). Geoscience Canada Volume 20 Number 1.

Palmero, P., Kern, F., Sommer, F., Lombardi, M., Gadow, R., & Montanaro, L. (2014). Issues in nanocomposite ceramic engineering: focus on processing and properties of alumina-based composites. *Journal of Applied Biomaterials & Functional Materials*, *12*(3), 113–128.

Pierson, H. O. (1993). *Handbook of Carbon , Graphite , Diamond and Fullerene*. (Pennsylvania State University, Ed.). New Mexico: Noyes Publications.

Prabu, M., Ramadoss, G., Senthilkumar, C., Magibalan, S., & Senthilkumar, P. (2016). Electrodischarge Machining of Al-TIB 2 Composite with and without Graphite Powder Suspended Dielectric, *11*(2), 1242–1244.

R.German. (1997). *Powder Metallurgy Science* (2nd ed.). The Pennsylvania State University.

Radhika, N., Subramanian, R., & Prasat, S. V. (2011). Tribological Behaviour of Aluminium / Alumina / Graphite Hybrid Metal Matrix Composite Using Taguchi ' s Techniques, *10*(5), 427–443.

Roy, A. K., Schulze, S., Hietschold, M., & Goedel, W. A. (2011). Oxidation protection of carbon fibers by coating with alumina and / or titania using atomic layer deposition. *Carbon*, *50*(3), 761–770.

Sabzevari, M., Abdolkarim, S., & Moloodi, A. (2015). Physical and mechanical properties of porous copper nanocomposite produced by powder metallurgy. *Advance Powder Technology*, 5–11.

Sivakumar, S., Sibu, C. P., Mukundan, P., Pillai, P. K., & Warrier, K. G. K. (2004).

Nanoporous titania – alumina mixed oxides — an alkoxide free sol – gel synthesis, *58*, 2664–2669.

Tamashausky, A. V. (2006). An Introduction to Synthetic Graphite . Introduction to Synthetic Graphite.

Thakur, S. K., Srivatsan, T. S., & Gupta, M. (2007). Synthesis and mechanical behavior of carbon nanotube-magnesium composites hybridized with nanoparticles of alumina. *Materials Science and Engineering A*, *466*(1–2), 32–37.

Vijayaraghavan. (2007). Machining Of Composites An Overview. *Machining of Composites an Overview*, *1*(1), 16–23.

Vikas Somani. (2006). Alumina-Aluminum Titanate-Titania Nanocomposite : Synthesis , Sintering Studies , Assessment of Bioactivity and, 37–38.

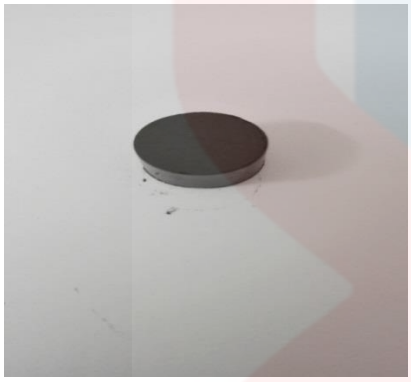
Wang, G., Yang, J., & Jiao, X. (2016). Materials Science & Engineering A Microstructure and mechanical properties of Ti – 22Al – 25Nb alloy fabricated by elemental powder metallurgy. *Materials Science & Engineering A*, *654*, 69–76.

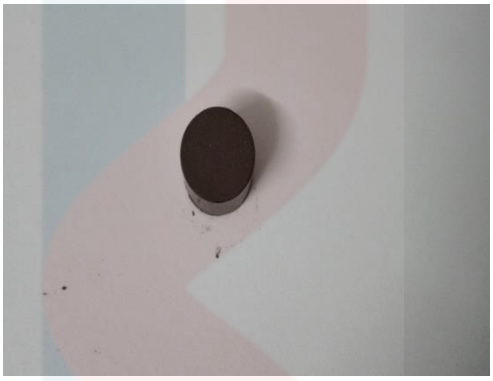
Zuhailawati, H., & Mahani, Y. (2009). Effects of milling time on hardness and electrical conductivity of in situ Cu – NbC composite produced by mechanical alloying, *476*, 142–146.

APPENDIX

Appendix 1

Compaction images





Milled powder

Appendix 2

Densification parameter

Time (h)	Pressure (MPa)	Densification parameter
15	200	0.155
	400	0.186
	600	0.188
	800	0.199
30	200	0.179
	400	0.192
	600	0.202
	800	0.206
45	200	0.192
	400	0.211
	600	0.213
	800	0.218
60	200	0.188
	400	0.188
	600	0.192
	800	0.195

Appendix 3

Example of compaction pressure calculations:

d= 10mm r = 5mm

Pressure: $P = \frac{F}{A} = \frac{F}{\pi r^2}$

F = $P \times A$

= $P\pi r^2$

= $(200)\pi(5)^2$

= 1.6 tons

Pressure (MPa)	tons
200	1.6
400	3.2
600	4.8
800	6.4

Appendix 4

Green density

Time (h)	Pressure (MPa)	Thickness (mm)	Diameter (mm)	Mass (g)
15	200	6.09	9.84	1.0476
	400	5.33	10.24	0.9345
	600	5.75	10.30	1.0659
	800	11.25	10.23	1.0446
30	200	6.00	10.29	1.0670
	400	5.76	10.27	1.0760
	600	5.53	10.22	1.0685
	800	5.41	10.23	1.0637
45	200	5.97	10.21	1.0824
	400	5.50	10.18	1.0794
	600	5.68	10.17	1.1364
	800	5.49	10.19	1.0721
60	200	5.95	10.18	1.0809
	400	5.85	10.13	1.0810
	600	5.92	10.20	1.0742
	800	5.86	10.18	1.0787