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***Effect of heat treatment on some mechanical
properties of Aluminum-Boron carbide
composites***

A thesis

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Abstract

Aluminum matrix composites reinforced with boron carbide are currently employed in the nuclear industry. It's used as a control rod in nuclear reactors; Since B_4C has very high absorption cross section for thermal neutrons. The objective of this work is to study the effect of heat treatment on the mechanical properties of the Al- B_4C metal matrix composites prepared by powder metallurgy techniques. Compressibility was measured with the Al powder. The chosen compaction pressure was 2500psi. Samples were prepared by adding 5%, 10%wt B_4C to Al as well as the Al powder. Sinterability was evaluated at different temperatures (400,450,500,550,600,650°C) during 2, 4, 6, 8; 10 hr for soaking time.

Mechanical test were evaluated, showed little improvement in hardness, compressive strength and young's modulus in temperatures 400,450,500,550°C, and significant improvement in hardness and compressive strength in temperatures 600and 650°C, and also improvement to the elastic modulus. Specific samples were irradiated by neutron source, and mechanical tests showed little decrease in their mechanical properties (hardness and compressive strength) due to low neutron fluence available that gives insignificant effect.

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List of symbols

Symbol	Definitions	Units
Al	Aluminium	-
B ₄ C	Boron Carbide	-
MMC _s	Metal Matrix Composites	-
PMC _s	Polymer Matrix Composites	-
	Compressive strength	N/m²
	Compressive strain	-
ρ _g	Green density	(gm/cm³)
	Calculated density	(gm/cm³)
VH	Vicker Hardness	-
psi	Pound per square inch	Psi=6.89*10³Pa
P	Pressure	N
F	Load	kg
d	Length of the side diagonals	mm

Chapter One

Theoretical Part

1.1 Introduction

The volume and number of applications of composite materials have grown steadily, penetrating and conquering new markets relentlessly. Modern composite materials constitute a significant proportion of the engineered materials market ranging from everyday products to sophisticated applications. While composites have already proven their worth as weight-saving materials, the current challenge is to make them cost effective. The efforts to produce economically attractive composite components have resulted in several innovative manufacturing techniques currently being used in the composites industry. It is obvious, especially for composites, that the improvement in manufacturing technology alone is not enough to overcome the cost hurdle. It is essential that there be an integrated effort in design, material, process, tooling, quality assurance, manufacturing, and even program management for composites 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 the sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years [1].

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 microfailure 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 [2].

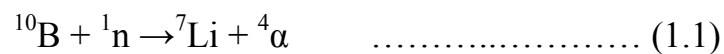
1.2 Neutron absorber materials

Neutron absorber materials consist of a chemical form of neutron absorber nuclide embedded in a matrix of metal or others materials. It's comprised of compound, alloy or a composite material that serves as a matrix to contain a neutron absorber nuclide. A neutron absorber nuclide is a nuclide that has a large thermal or epithermal neutron absorption cross section or both [3].

Boron is a useful control material for control rod to many kinds of reactors. The very high thermal absorption cross section of ^{10}B and the low cost of boron have led to wide use of boron-containing materials in control rods and burnable poisons for thermal reactors. The absorption cross section of boron is large over a considerable range of neutron energies, making it suitable for not only control materials but also for neutron shielding [4]. ^{10}B having a large thermal neutron absorption cross section, in excess of 3,800 barns, the cross section of ^{10}B diminishes with increasing neutron energy and varies as $1/v$ with increasing neutron velocity; where v is the neutron velocity, i.e. proportional to its kinetic energy.

Boron is nonmetallic and is not suitable for control rod use in its pure form. For reactor use, it is generally incorporated into a metallic material. Two of such composite materials are described below.

- Stainless-steel alloys dispersions with boron have been employed to some extent in reactor control. The performance of boron-stainless-steel materials is limited because of the $^{10}\text{B} (n, \alpha) \text{Li}$ reaction as in the following reaction



The absorption reaction is a transmutation with the α -particle produced becoming a helium atom. The production of atoms having about twice the volume of the original atoms leads to severe swelling; hence these materials have not been used as control rods in commercial power reactors.

- The refractory compound boron carbide (B_4C) has been used as a control material dispersed in aluminum (boral). Boral, is technically a cermet. A cermet is produced from an ingot that is an aluminum clad container filled with a homogenous mixture of boron carbide powder and atomized aluminum powder. When the ingot is heated and subsequently hot rolled to plate form, the aluminum particles sinter at the rolling temperature and under the pressure caused by the rolling process. However, the sintering process is not complete in that the core of the final product contains some porosity [4].

Aluminum has been the most commonly used, although stainless steel has also been used extensively. Aluminum matrix absorbers can be manufactured to final form (generally a thin plate) by a variety of methods. In one method, a preform with near maximum theoretical density can be produced by casting, followed by

hot rolling to final gage. In another, a fully dense cylindrical billet of aluminum and boron carbide is produced using powdered metallurgy technology. These materials, commonly known as Metal Matrix Composites (MMCs), are first extruded and cut to a preform, and then hot rolled to final thickness.

The solubility of boron in aluminum is very low and that only limited quantities of boron can be alloyed with aluminum. Boron forms boron-rich borides that precipitate at the grain boundaries of the aluminum alloy matrix. Boron-10 is usually incorporated as an intermetallic boride phase (e.g. AlB_2) either in an aluminum alloy or stainless steel; or as a stable chemical compound such as boron carbide (B_4C), typically in aluminum Metal Matrix Composites (MMC) or cermet because of the relatively low density of aluminum, small percentages of naturally occurring boron in an alloy with aluminum have relatively low boron-10 areal densities [3].

The development of nuclear power engineering requires low-density structural materials to perform at high temperatures and loads and to have a large effective thermal-neutron capture cross-section. The Al- B_4C composites consider much promise. Aluminum and boron and their compounds have low density. The effective thermal neutron capture cross-section of aluminum is small (23 barns) and of boron is large (760 barns) [5].

1.3 powder metallurgy

Powder metallurgy may be defined as the art of producing metal powders and using them to make serviceable objects. Powder metallurgy principles 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 commonplace during the middle ages [6].

Modern powder metallurgy (P/M) technology commenced in the 1920s with the production of tungsten carbides and the mass production of porous bronze bushes for bearings. During the Second World War, further developments took place in the manufacture of a great variety of ferrous and non-ferrous materials, including many composites, and there was a steady development during the post-war years until the early 1960s. Since then the growth of the Powder Metallurgy (P/M) has expanded more rapidly, mainly for three reasons, economical processing, unique properties, and captive processes. Figure (1.1) shows the fabrication process of composites by using powder metallurgy techniques [7, 8].

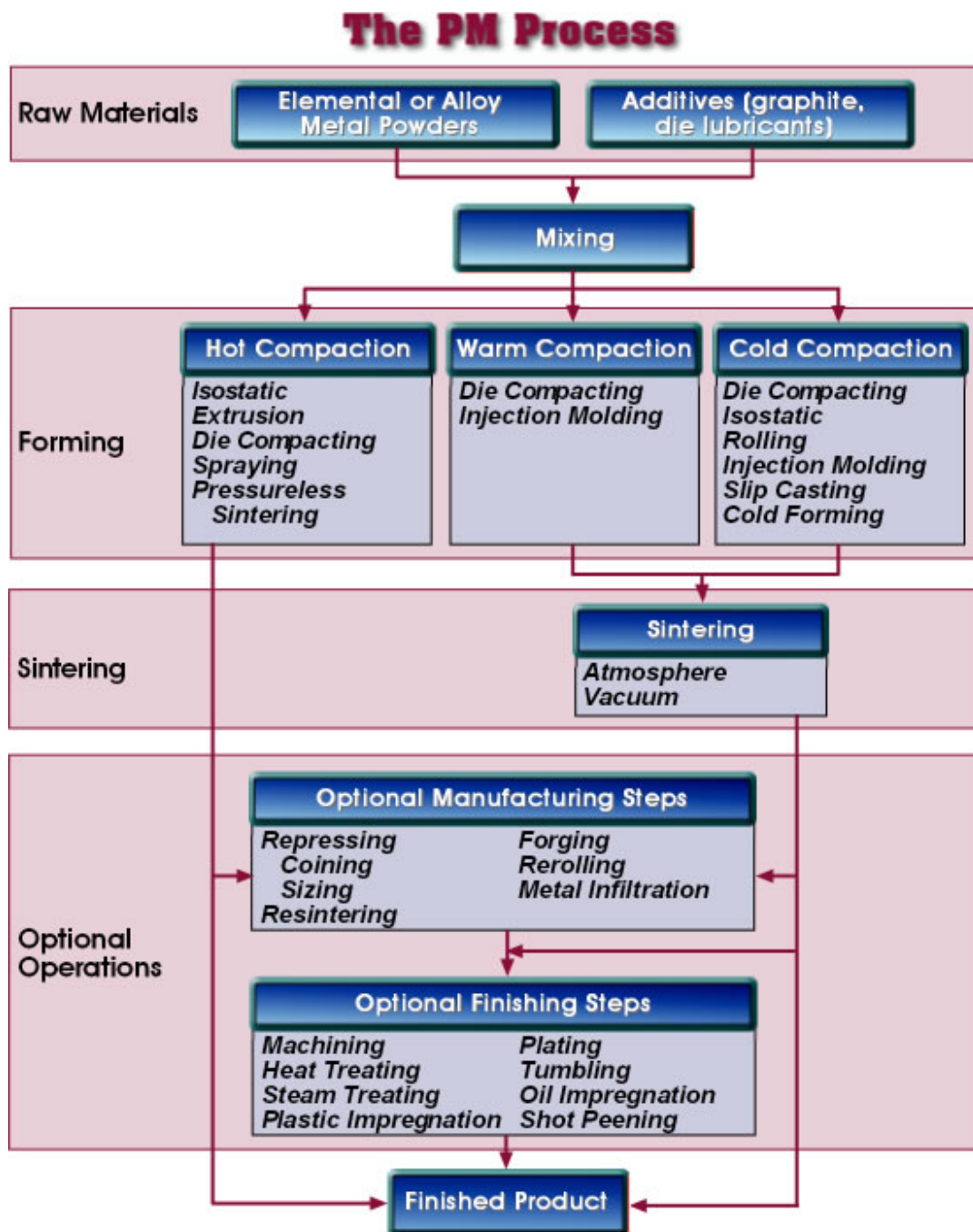


Figure (1.1) shows fabrication of composites by powder metallurgy techniques [8].

1.3.1 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.

Many mechanical and chemical methods are used to produce powders for specific applications, but the three most important methods are atomization, reduction of oxide, and electrolytic deposition. 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].

1.3.2 powder compaction

The most important operation in powder metallurgy is compacting or pressing. The ability to obtain a satisfactory pressed density often determines the feasibility to manufacture by powder metallurgy. Most compacting is done cold, although there are some applications for which compacts are hot pressed; hot pressing involves applying pressure and temperature simultaneously so that molding (compacting) and sintering of the powder takes place at the same time in the die. The disadvantage of hot pressing as compared with cold compacting and sintering are a reduction in gas content, and shrinkage effect, along with higher strength, hardness, elongation, and density. Hot pressing is used only to a limited extent, primarily for the production of very hard cemented-carbide parts. The principle disadvantage of this method is the high cost of dies to stand up under pressure at elevated temperature [6].

The compaction of metal powders has the following major functions:

- To consolidate the powder into desired shapes

- To impart, as much as possible , the desired final dimensions with due consideration for any dimensional change resulting from sintering
- To give the desired level and type of porosity
- To provide adequate strength for subsequent handling [7].

Compacting techniques may be classified into two types

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

Die compacting is the most widely used method. The useful sequence of operations in die compaction consists in filling the die cavity with a definite volume of powder; application of the required pressure by the movement of the lower and upper punches toward each other , and finally ejection of the green compact by the lower punch. These operations are shown schematically in figure (1.2)

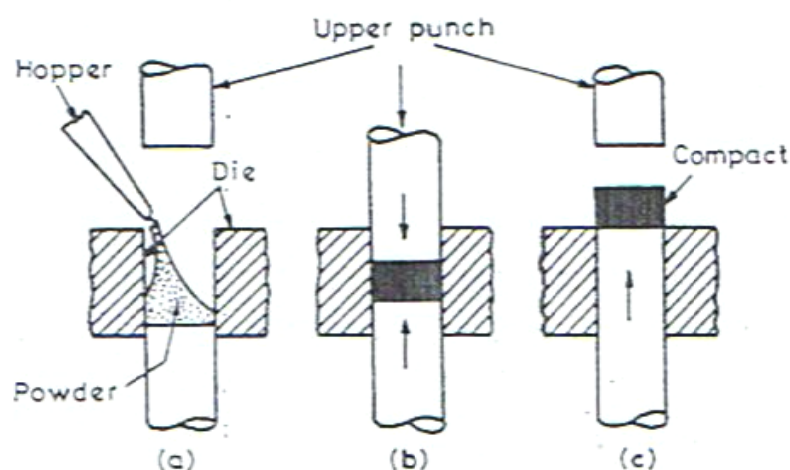


Figure (1.2) shows the die pressing [9].

The pressures commonly employed range from 19 to 50 tons/sqin. The pressure may be obtained by either mechanical or hydraulic presses. Mechanical presses are available with pressure ratings of 10–150 tons and speed of 6 to 150 strokes/min. The important features of mechanical presses are high speed production rates, flexibility in design, simplicity and economy in operation, and relatively low investment and maintenance costs. Hydraulic presses have higher pressure ratings, up to 5,000 tons but slower stroke speeds, generally less than 20 min. These presses are used for higher pressure, more complicated powder metal parts. Dies are usually made of hardened, ground, and lapped tool steels. When the powder to be compacted consists of hard abrasive particles, the die is generally constructed of two parts. The tough outer section supports the hardened, polished, wear resistance insert which is working surface of the die. These replaceable liners are discarded when worn and reduce the cost of the die upkeep. The punches are made of die steel heat-treated to be slightly softer than the die, since they are usually easier to replace than the die. They must be perfectly aligned and very closely fitted [6].

1.3.3 Heat treatment and Sintering 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

- Properties, e.g., better machinability, improved ductility, and homogenous structure.
- Conditions e.g., that of relieved 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 [9].

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 [9].

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:

- Initial bonding among particles

- Neck growth
- Pore channel closure
- Pore rounding
- Pore shrinkage
- Pore coarsening

Figure (1.3) 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 leads 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.

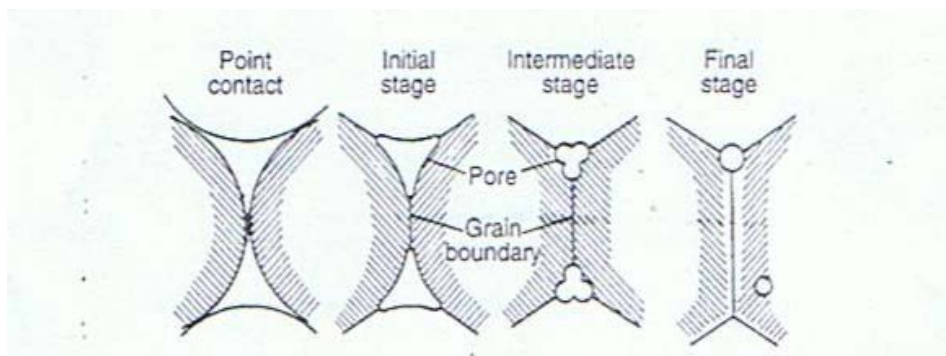


Figure (1.3) shows various sintering stages [7].

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 [7].

1.4 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 [5].

As defined by Jartiz, [10] Composites are multifunctional material systems 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.

Kelly [11] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength or resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Berghezan [12] 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 [13] 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.

1.4.1 Characteristics of the composites

The combination of dissimilar materials can have unique and very advantageous properties if the materials have appropriate characteristics, and result in a material that 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 [14].

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.

Concentration, usually measured as volume or weight fraction, determines 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 [5].

1.4.2 Classification of the composites

Composite materials can be classified in different ways:

- Classification based on the nature of matrix material into [8]:
 1. Metal Matrix Composites MMC_S
 2. Ceramics Matrix Composites CMC_S
 3. Polymer Matrix Composites PMC_S
- 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 figure (1.4). The two broad classes of composites are [1]:
 - Particulate composites
 - Fibrous composites

1.4.2.1 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[1].

1.4.2.2 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 [1].

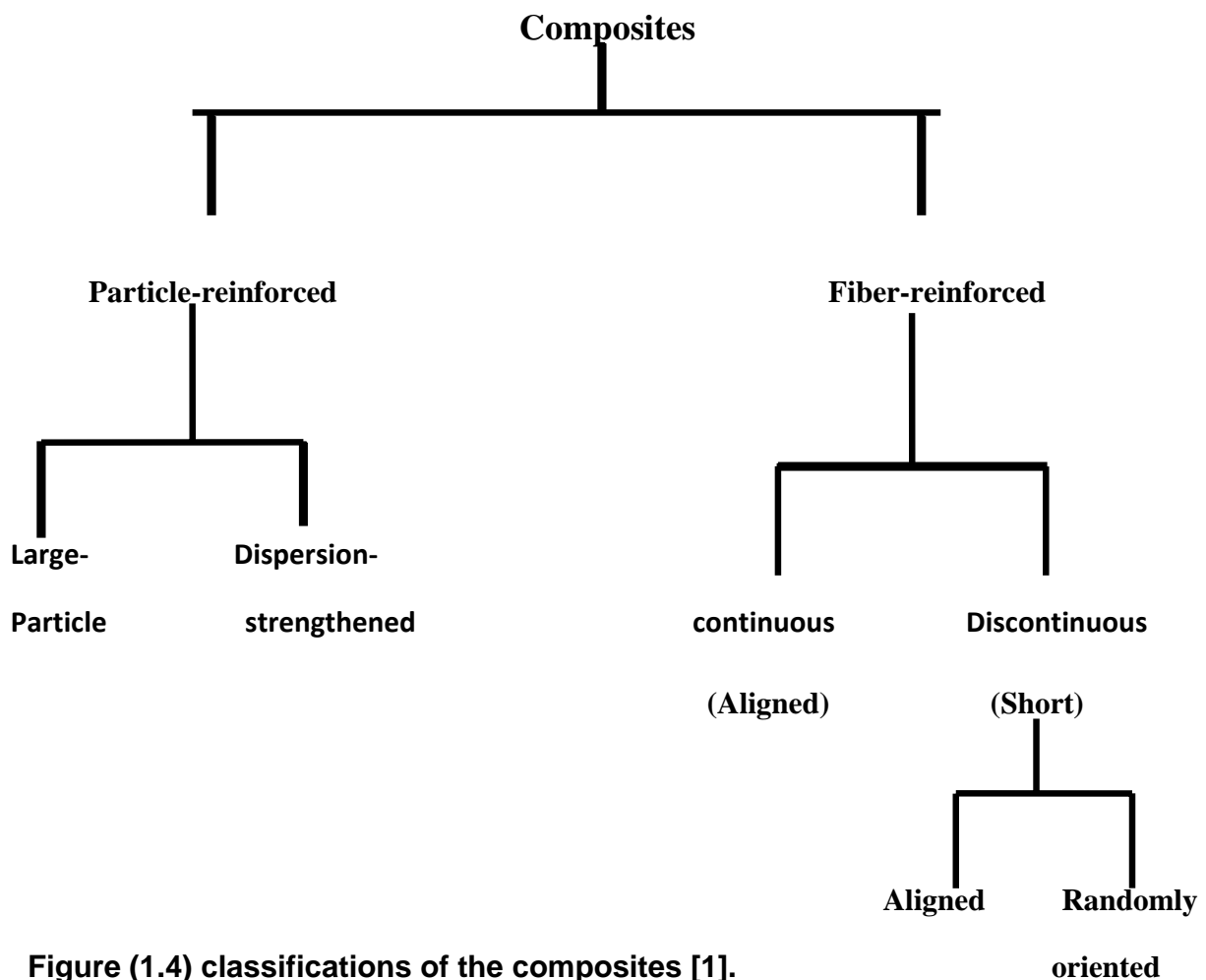


Figure (1.4) classifications of the composites [1].

1.4.3 Metal Matrix Composite (MMC)

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 [5].

The superalloys, 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 [15].

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 [16]

- 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 fabricability 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 [5].

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. For example aluminum alloys are chosen 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, B₄C, nitrides, and Al₂O₃ are in the form of continuous fibers, whiskers, platelets, or particles [17].

1.4.4 Aluminum–boron carbide system

The significant drawback with ‘aluminum metal and alloys as structural materials is their relatively low elastic modulus. This make them elastically ‘softer’ compared to common structural materials like steel. As a consequence, such light-weight alloys tend to experience greater deflection under load. Excessive deflections under load are undesirable from a design aspect and prevent the use of these materials in many load-bearing weight-sensitive applications. One strategy to stiffen these materials is through the incorporation of particles to form a metal matrix composite (MMC). Candidate reinforcing particles are those which have a high stiffness and low density [18].

Boron carbide is a very attractive material. It has low density (2.51 g/cm^3), excellent chemical resistance and extreme hardness that make boron carbide ideal for a variety of structural and functional applications. Boron carbide (B_4C) also has a high neutron absorption cross-section making it a valuable material for nuclear applications [19]. It's also one of the few elements that do not emit gamma radiation after being irradiated and helium is released. Thus, boron is uniquely suited for nuclear applications although it has not good mechanical properties. Therefore, it's used alloyed with low or medium cross-section materials, such as iron or aluminum. Due to its lower cost, B_4C is also employed [20].

Aluminum base material is readily available, light-weight, and under reasonable processing conditions, reactive to B_4C . Theoretically, the elastic modulus of an aluminum alloy MMC can be increased significantly, even at a boron carbide concentration of 10% by volume. Hence B_4C –Al composites have the potential to combine the high stiffness and hardness of B_4C with the ductility of Al without defeating the goal of obtaining a stiff low density material. Suggested applications for B_4C –Al composites include their use as structural neutron

absorber, armor plate materials, and as a substrate material for computer hard disks. Compared to other MMCs the experimental work on B₄C–Al composites for structural application is scattered and limited [18].

1.5 Mechanical properties of materials

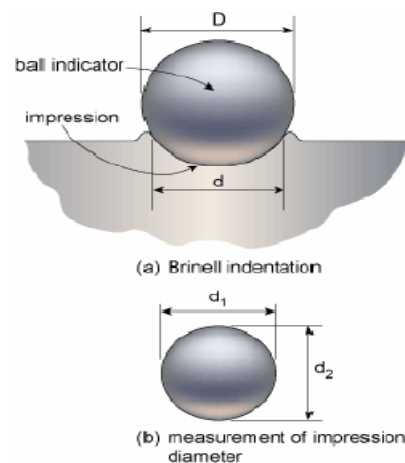
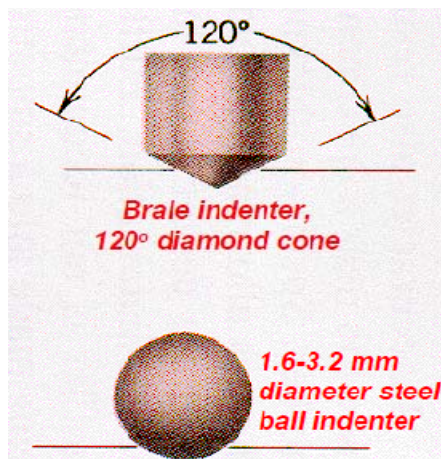
Mechanical properties include those characteristics of material that describe its behavior under the action of external forces. The response of the materials to applied forces will depend on the type of bonding, the structural arrangement of atoms or molecules and the type, the number of imperfections, 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].

1.5.1 Hardness

Hardness is the measure of the material's resistance to localized plastic deformation (e.g. dent or scratch) [9]. The hardness of materials depends upon the type of bonding forces between atoms, ions, or molecules and increases with the magnitude of these forces. The hardness of materials is also increased by alloying, cold work and precipitation hardening.

A qualitative and somewhat arbitrary hardness indexing scheme was devised, termed the Mohs scale, which ranged from 1 on the soft end for talc to 10 for diamond. Quantitative hardness techniques have been developed over the years in which a small indenter is forced into the surface of a material to be tested, under controlled conditions of load and rate of application. The depth or size of the resulting indentation is measured, which in turn is related to a hardness number;

the softer the material, the larger and deeper the indentation, and the lower the hardness index number. Measured hardnesses are only relative (rather than absolute), and care should be exercised when comparing values determined by different techniques. Different types of quantitative hardness test have been designed (Rockwell, Brinell, Vickers, etc.). Usually a small indenter (sphere, cone or pyramid) is forced into the surface of a material under conditions of controlled magnitude and rate of loading. The depth or size of indentation is measured. The tests somewhat approximate, but popular because they are easy and non-destructive (except for the small dent).



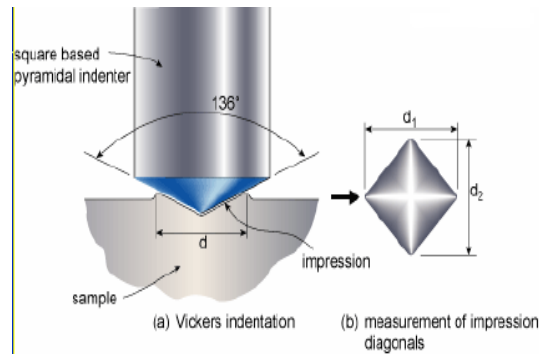


Figure (1.5) shows types of indenters [15].

The vickers test consist in impressing a pyramidal diamond penetration into the surface of a test piece [21]

Applied loads are much smaller than Rockwell and Brinell, ranging between 1 and 1000 gm [15]. Loads are applied for periods 10–15 s for ferrous metals and 30 ± 2 s for non-ferrous metals, after which the load is removed and two diagonals of the indent are measured [22]. careful specimen surface preparation (grinding and polishing) may be necessary to ensure a well defined indentation that may be accurately measured [15]. The vicker hardness number HV is the ratio of the load to the area of the side surface of the indent which is easily expressed through the length of the diagonal

$$HV = 2p \frac{\sin\left(\frac{\alpha}{2}\right)}{d^2} \dots \dots \dots (1.1)$$

Where α is the angle between the opposite faces at the pyramid tip, d is the arithmetic mean of two diagonals.

The surface of the piece for the Vicker hardness tests should correspond to the 10th class of surface finish. the smallest allowable radius of surface curvature is 5 mm. the distance from an indent to the edge of a test piece or to an adjacent indent

must be at least 2.5 times the length of the diagonal. The minimal thickness of a test piece or layer should be 1.2 times the indent diagonal for steel and 1.5 times the diagonal for non-ferrous metals [21].

1.5.2 Compressive strength

Compressive strength is the ability of a metal to withstand pressure acting on a given plane.

The compressive stress

$$\sigma = F/A \dots\dots\dots (1.2)$$

Where F is the load applied perpendicular to specimen cross section, A_0 is the cross sectional area (perpendicular to the force) before applications of the load.

The compressive strain

$$\varepsilon = \Delta L/L_0 \dots\dots\dots (1.3)$$

Where ΔL is change in length, L_0 is the original length [15].

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. This can be explained by that at small deformation the friction at the end faces of the test piece is still ineffective. In view of the complexity of the methods (the use of strain gages is indispensable), these characteristics are only seldom determined

in compressive tests. The transition of the fracture in compression differs substantially from the observed in tension. Many brittle materials (cast iron, casting aluminum alloys) which exhibit brittle fracture by rupture in tension, can fail by shear in compression [21].

The compression test ranks low on the list of routine acceptance test for metals, it can be used to obtain useful data in such fields as plastic or ceramics. The compression test is not used for most metals because it's not as reliable an indicator of ductility as are the tensile test and reduction of the area test [8].

1.6 Effect of Neutron irradiation on material

Neutrons have energies spreading over a very wide range, since the fission neutrons with initial energies of about 1 to 15 MeV may be slowed down by moderator until their energies are in their thermal range, 0.05 eV. Neutrons with energies between the fission and thermal range are called epithermal neutrons [22].

The principal effect of radiation on metals and alloys is the creation of lattice vacancies and interstitial atoms in an otherwise perfect crystal. This results in an overall dilation that decreases the density of the material. In metals that were neutron-irradiated at ambient temperature, the measured decrease in density was much smaller than that predicted by theory [23].

About 20 eV of energy is needed to displace a lattice atom in a metal producing an interstitial atom and a vacancy. Consequently, when neutrons with energies in Mev range enter a metal, most collisions they make displace a lattice atom with great vigour, even through transference of energy is inefficient the energy transferred may be estimated by simple mechanics, as for collisions between billiard balls, and the amount transferred depends on the atomic mass. The typical

primary “knock-on” as it’s called, produced by a 2 MeV neutron therefore has enough energy to displace about 1000 further atoms , the secondary”knoch-ons”, as they are called. Each primary or secondary knock-on must leave a vacancy behind it. Whereas the primary knock-ons hit by a single neutron are widely spaced (some centimeters apart) because the neutrons, being uncharged, only make a collision when they hit an atom’s nucleus, the primary knock-ons themselves make very frequent collisions since they move slowly enough for each other’s electron shells to be effective barriers, and the secondaries are densely clustered. There are several uncertainties about the clusters of damage. [22].

Contrary to theoretical analyses that predicted large modifications to elastic properties, tests have shown that the elastic moduli of metals are not appreciably affected by neutrons below a fluence of 10^{17} n/cm². Plastic properties of metals are markedly affected by radiation. The properties affected include yield strength, ultimate tensile strength, and elongation, reduction in area, creep, rupture stress, fatigue stress, hardness, impact strength, and ductile-to-brittle transition temperature. In general, metals exhibit reduced plasticity and ductility and increased hardness following irradiation. As a possible explanation of the foregoing observations, it has been suggested that because plasticity is associated largely with the motion of dislocations, any mechanism that impedes this motion can produce the class of effects observed in irradiated metals.

Tests conducted to determine the effects of neutron irradiation on the mechanical properties of metals and alloys have shown that temperature of exposure, time at temperature, fluence, energy spectrum, and material properties (i.e., composition, degree of cold work, prior heat treatment and quenching, and grain size) are important variables [23].

1.7 Literatures review

Development of composite materials based on boron carbide and aluminum is of interest primarily for the fact that the two components exhibit a low density with the additional advantageous features that boron carbide has a very high hardness and aluminum and its alloys normally have an enhanced ductility. These advantageous features are undoubtedly attractive in the production of high-strength materials such as armor. A promising method for preparing boron carbide-based materials impregnated with aluminum has been proposed in which the porous cage of B_4C is infiltrated with aluminum [24].

Halverson et al, in 1989[25] investigated the processing of aluminum–boron carbide composite. The processing problems associated with boron carbide and the limitations of its mechanical properties can be significantly reduced when a metal phase (e.g., aluminum) is added. Lower densification temperatures and higher fracture toughness will result. Based on, reaction thermodynamics, and densification kinetics, they have established reliable criteria for fabricating Al– B_4C particulate composites. Because chemical reactions cannot be eliminated, it is necessary to process Al– B_4C by rapidly heating to near $1200^\circ C$ (to ensure wetting) and subsequently heat-treating below $1200^\circ C$ (for microstructural development).

Beidler et al, in 1992[26] studied development of Al – B_4C cermet for use as an improved structural neutron absorber. The cost of this cermet can be expected to be much lower than hot-pressed boron carbide material. Properties were measured over a range of loadings and particle distributions of the boron carbide.

Bond and Inal, in 1995[27] studied shock-compacted aluminum–boron carbide composites. The explosive consolidation can be used as a processing route to

produce metal-matrix composites, comprising an aluminum-based matrix and boron-carbide reinforcement. Composites containing 30, 50 and 70 vol% boron carbide–aluminum-alloy matrix were successfully compacted at 10–12 GPa pressure. It is predicted that the high density of dislocations produced during the compaction will lead to accelerated aging in age-hardenable aluminum alloy matrices.

Pyzik et al, in 1995[28] made a study about the phase development of Al–B–C and effect on mechanical properties of Al–B₄C derived composites. They concluded that the highest hardness (88 Rockwell A) and Young's modulus (310 GPa) are obtained in Al₄BC-rich samples. AlB₂-containing samples exhibit lower hardness and Young's modulus but higher fracture toughness. While the modulus, Poisson's ratio, and hardness of multiphase Al–B₄C composites containing 5-10 vol% free metal are comparable to ceramics, the unique advantage of this family of materials is low density (<2.7 g/cm³) and higher than 7 MPa·m^{1/2} fracture toughness.

Viala et al, in 1997[29] studied chemical reactivity of aluminum with boron carbide at temperatures ranging from (627–1000⁰C). Concluded that the temperature in the investigated range, B₄C has been observed to react with solid or liquid Al. As long as the temperature is lower than 660⁰C, i.e. as long as Al is in the solid state, interaction proceeds very slowly, giving rise to the formation of ternary carbide (Al₃BC) and to diboride (AlB₂). At temperatures higher or equal to 660⁰C, Al is in the liquid state and the reaction rate increases sharply. Up to 868±4⁰ C, the reaction products are Al₃BC and AlB₂.

Chapman et al, in 1999[30] studied wear resistance aluminum–boron carbide cermets for automotive brake applications. Concluded that the resulting friction

coefficients fell within the industry standard range of 0.3 to 0.45 after break-in and did not change with temperature. Optical Metallography (OM), and Scanning Electron Microscopy (SEM) analysis indicated that wear surfaces were coated with an iron-rich, two phase glazing, with one phase containing substantial amounts of aluminum. This interface glazing is believed to enhance performance through maintenance of smooth surfaces and increased pad to rotor contact.

Palfey et al, in 2000[31] evaluated the mechanical response of boron carbide–Aluminum composites during compressive loading. The compressive stress-strain and failure behavior of 80vol%B₄C–Al and 65vol%B₄C–Al composites has been examined at room temperature and at strain rates of 10⁻⁴ and 10⁻³s⁻¹. The compressive fracture strength of the composite is more than a factor of two greater than that of the B₄C skeleton with fracture stresses in excess of 2000 MPa in the 65vol% B₄C–Al and greater than 3800 MPa in the 80vol% B₄C–Al. The characteristics of the compressive stress-strain response as well as the ratios of transverse strains to longitudinal strain indicate that failure of the 65vol% B₄C–Al composite is controlled by the Al, but that the 80vol%B₄C–Al is dominated by the B₄C.

Arslan et al, in 2003[32] made a study about the quantitative x-ray diffraction analysis of reactive infiltrated aluminum–boron carbide composites. Concluded that AlB₂ phase forms at relatively small amounts and its formation can be significantly suppressed or totally eliminated by increasing the particle size of the starting B₄C powder and/or altering its surface chemistry.

Zhang et al, in 2004 [33] studied the high strain rate response of Al–B₄C composites. Al–B₄C Metal-Matrix Composites (MMC). The MMC fabricated by two different powder consolidation routes, extrusion and sintering/Hot Isostatic

Pressing (HIPing), were made and tested over a wide range of strain rates (10^{-4} to 10^4 s^{-1}). The strength of these MMCs increases with increasing volume fraction of particulate reinforcement. Strain hardening is observed to increase with increasing volume fraction of reinforcement at lower strains (<5%), but tends to be insensitive to volume fraction at higher strains. The composites show significant strain rate dependence. The fabrication route affects the strength of the matrix material, as reflected in the microstructure, and this effect carries on into the corresponding composites.

Lillo, in 2005[34] conducted enhancing ductility of AL + 10% B₄C through equal-channel angular extrusion processing. Evaluation of the mechanical properties showed little improvement to the elastic modulus, low tensile strength and no significant amount of ductility. Microstructural characterization revealed a redistribution of the B₄C particles. The biggest improvement was observed in the elongation to fracture which increased to >10%.

Kimmari and Kommel, in 2006 [35] made a studied about the application of the continuous indentation test method for the characterization of mechanical properties of Al–B₄C composites. The hardness and reduced elastic modulus of composites were estimated from the unloading part of the indentation curve. The composites should be tested in a wide range of indentation loads to estimate the transition from the load-dependent to load-independent hardness, which is related to the indentation brittleness of the material. Loading rate effect should be taken into account to adequately estimate mechanical properties of the composites.

Abenojar et al, in 2007[20] investigated optimization of processing parameters for the Al + 10% B₄C system obtained by mechanical alloying. Hardness was

evaluated at different temperatures. The best results were obtained at 635°C and argon atmosphere as sintering conditions.

Kerti and Toptan, in 2007[36] studied microstructural variations in cast B₄C-reinforced Aluminum Matrix Composites (AMCs) concluded that B₄C addition with bigger particle size resulted in better microstructure with free of agglomerated particles.

Hulbert et al, in 2008 [19] studied the modeling of spark plasma sintered, functionally graded aluminum–boron carbide composites. Melt infiltration, X-ray diffraction reveals that some of the defects are dissipated and small amounts of Al₃BC are present. Numerical modeling results indicate the presence of a 132°C temperature difference between the top and bottom of a specimen using this method and help confirm experimental observations.

Kimmari and Kommel, in 2008 [37] tested the influence of rapid temperature change on crack nucleation and propagation in lightweight Al–B₄C composite. The composite was produced using Self-propagating High temperature Synthesis (SHS) with subsequent heat treatment at 700°C in vacuum. Quenching in water was chosen as thermal loading. The specimens have been subjected to a specified number of thermal cycles and then analyzed. Postshocked samples have been characterized by means of microstructural and fractographical investigations to analyze the crack paths.

Mohanty et al, in 2008 [18] studied the fabrication and properties of aluminum reinforced boron carbide matrix composites. There was an 11-fold increase in hardness with 25 wt. % B₄C addition., the flexure modulus showed up to 8 times improvement with increased fraction of reinforcement. The tendency for

brittle fracture increased with higher reinforcement fractions, and it is concluded that even though uncoated boron carbide reinforcements embrittle the aluminum matrix in as-reinforced condition, improvement in other as discussed properties is significant. The study also indicates that the ductility of the Metal Matrix Composite (MMC) may be improved by increasing the interfacial bonding and decreasing the overall porosity contents of the composite.

Salvador et al, in 2009 [38] studied high temperature mechanical properties of aluminum alloys reinforced with boron carbide particles. The composites were produced by hot extrusion. The tensile properties and fracture analysis of these materials were investigated room temperature and at a high temperature to determine their ultimate strength and strain to failure. The surface was analysed by scanning electron microscopy. They observed that the mechanical properties has been improved by adding B₄C particles as reinforcement, and also the tensile strength of Aluminum Matrix Composites (AMCs) with B₄C decreases as the temperature increased.

1.8 The objective of this study

The objective of this work is to study the influence of heat treatment of aluminum based boron carbide metal matrix composite. The following physical characteristics have been measured

- Compact density of green and sintered samples
- Hardness
- Compressive strength
- Microstructure image
- Irradiation effect.

1.9 Thesis layout

Chapter one give the theoretical part which contain comprehensions about composite materials, mechanical properties and literature review.

Chapter two includes the experimental part that explains the types of materials (matrix and reinforcement materials) that are used to make samples for tests.

Chapter three presents the results and discussion about the differences resulting from adding boron carbide powder to aluminum with different weight percentage on the mechanical properties; hardness and compression.

In Chapter four the main conclusions that can be drawn from this work are given.

Chapter two

Experimental Methods and Procedures

2.1 Introduction

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

2.2 materials

1. Aluminum powder purity > 99%, particle size less than 100 micron, Fluka co. Switzerland.
2. Boron carbide, purity 99.6%, particle size less than 20 micron, Fluka co. Switzerland.

2.3 Apparatus

2.3.1 Die description

Two dies with different diameters (10 and 15mm) were used. These dies were made from carbon steel which is manufactured in our victories.

2.3.2 Press : electrical press, Carver, Inc, part No. 973147A

2.3.3 Furnace:

Carbolite Tube furnace, maximum temperature 1200°C supplied with eurotherm temperature controller model 818 Carbolite company, U.K

2.3.4 Optical microscopy:

The microscope type Nikon, Eclipse ME600 (Chapin) supplied with a camera type Nikon Digital Camera, DX1200X.

2.3.5 Microhardness tester, ADOLPH.I.Buehler.Inc, U.S.A 60204.

2.3.6 Compression tester, Tinius Olson Inc, U.K., maximum capacity 50 kN.

2.3.7 Neutron source:

An (Am–Be) source with flux density (5×10^5 n/cm².s) was used. It's emits fast neutrons from the (α , n) reaction such as:



This source consist of a rod of (Am–Be) surrounded by a paraffin wax. The paraffin wax is usually used for moderating the fast neutrons to thermal neutrons energies giving flux of density (5×10^3 n/cm².s) approximately.

2.4 Methods:

2.4.1 Sample preparation

To prepare a suitable sample for Al powder two types of dies with radius 10 and 15 mm were used with different weight 0.5,1,1.5,2,2.5,3gm.

The Al powder was pressed with ranges 500, 1000, 1500, 2000, 2500 psi to evaluate the height of samples. It was found that the proper sample height was 10mm.

- pure aluminum
- 95% Aluminum + 5% Boron carbide
- 90% Aluminum + 10% Boron carbide

Fifty grams for powder were mixed and blended to get homogenous mixture. Assuming 90% green density, the mould was polished with lubricant to prevent adhesion, 2.12 gm of the powder were poured inside the die hole, and subjected to a specific pressure using Uniaxial press techniques for five minutes, Then withdrawn from the die to get the green compacts .Such amount of powder should give a green compact with height about 10mm.

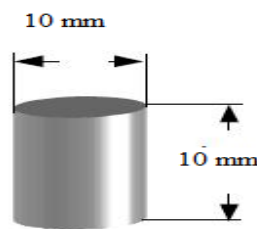


Figure (2.1) shows dimension of green compact as calculated assuming 90% at least relative density. .

2.4.2 Effect of particle size:

Aluminum powder of particle less than 100 micron was sieved using standard microsieve. The following fraction were collected, < 20 micron, 20–30 micron, 30–40 micron, 40–60 micron,60–80 micron,80–100 micron and >100 micron

2.4.3 Effect of heat treatment:

The green compacts were given the following heat treatment at ordinary atmosphere

- Temperature rise rate 240°C/h , soaking time 2hr at temperatures $400, 450, 500, 550, 600, 650^{\circ}\text{C}$
- Temperature rise 240°C/h , soaking time 2, 4, 6, 8, 10 hr at temperature 600°C
- Cooling rate 240°C/h

Figure (2.2) shows heat regime.

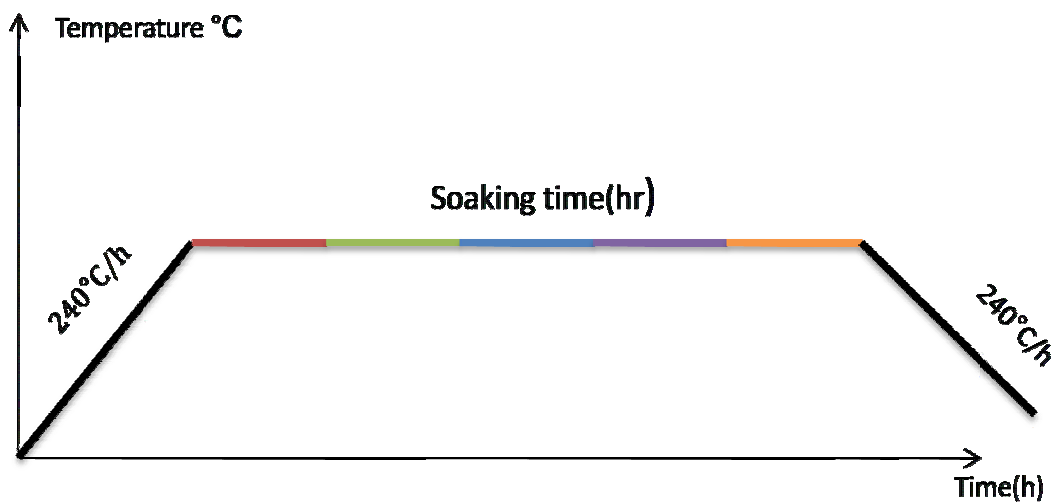


Figure (2.2) heat regime.

2.4.4 Density measurements:

The green density means density calculated after compacting process, before sintering. Green density has been calculated from the following equation

$$\rho_g = M_g / V_g \dots \dots \dots (2.2)$$

Where:

ρ_g : green density (gm/cm³)

M_g : sample weight after compact

V_g : sample volume

Calculated density for the mixture of aluminium and boron carbide have been calculated from the equation

$$\rho_c = 100 * \frac{\rho_1 \rho_2}{x_1 \rho_2 + x_2 \rho_1} \dots \dots \dots (2.3)$$

where:

ρ_c : calculated density for the composites (gm/cm³)

ρ_1 : Aluminum density is 2.69 gm/cm³

ρ_2 : Boron carbide density 2.5 gm/cm³

x_1, x_2 : weight percent for aluminium and boron carbide respectively.

$$\text{True density } \rho = M/V \dots \dots \dots (2.4)$$

ρ : density (gm/cm³)

M: sample weight after compact

V: sample volume

Vernier with accuracy of 0.01 mm was used to measure height and radius of the compacts.

2.4.5 Irradiation method

Samples prepared with Pure Al, Al+5%B₄C, Al+10%B₄C with average particle size 50µm heat treated at temperatures 400,450,500,550,600°C were exposed to neutron source.

The samples were covered with aluminum foil and placed at paraffin wax at a distance of (5 cm) from the neutron source (Am–Be) with thermal flux density (5000 n.cm⁻² .s⁻¹). The irradiation time was 75 days with fluence of thermal neutron (3.24 x10¹⁰ n.cm⁻²).

2.5 Material tests

2.5.1 Hardness test

Hardness test was carried out using Vickers hardness test. Vickers hardness test uses a diamond indenter of square pyramid shape which applied to the surface of specimen for (10-15) sec and 500gm load after refining with 1000 grit grinding silicon carbide paper in order to reduce the machining scratches and decrease the influence of surface defects on hardness behavior.

Table (2.1) shows hardness test results before and after irradiation.

	Temperature (°C)	Hardness before irradiation	Hardness after irradiation
Al	400	18.85	23.3
	450	19.48	25.85
	500	21.66	26.1
	550	22.9	26.95
	600	25.25	28
Al+5%B₄C			
	400	33.2	29
	450	38.45	35.2
	500	41.54	38.23
	550	45.87	42
	600	52.62	48.72
Al+10%B₄C			
	400	57.62	52.2
	450	63.88	56.33
	500	68.81	59
	550	75.59	71.35
	600	87.98	83

2.5.2 Compression test

The compression test involves an axial compression load being applied to a standard specimen of circular cross section with speed 0.05 mm/min and this causes the specimen to be compressed.

Table (2.2) shows compression test results.

	Temperature (°C)	Compressive strength
Al	400	40.7
	450	48.3
	500	53.5
	550	58
	600	61.6
	650	73.88
Al+5%B₄C	400	73.88
	450	76.43
	500	78.98
	550	84.07
	600	89.17
	650	119.3
Al+10%B₄C	400	91.7
	450	94.2
	500	99.36
	550	114.64
	600	133
	650	153

Chapter Three

Result and Discussion

3.1 Introduction

This chapter presents the results of this study and is discussed to explain the differences resulting from adding boron carbide powder with different weight fractions on the mechanical properties; compression, and hardness compared with pure Al matrix.

3.2 Particle size analyses of the aluminium powder:

The behavior of metal powders during processing depends upon the particle size distribution, particle shape and structure of powders.

In sieving, the powder is shaken through a woven wire mesh screen with square holes. The particle size is defined by the apertures of the sieve which retains a given particle.

A specific amount of Al powder was screened using standard microsieves. The following result of screen test was obtained as shown in table (3.1).

From this table, the results shown in figure (3.1) the average or statistically its particle size is calculated using cumulative density function CDF, which is equal to $50\mu\text{m}$ [40]. The result of this analysis is shown in figure (3.2).

Table (3.1) values of particle size, average particle size, weight percent, and cumulative weight percent.

Particle size(μm) range	Average particle size(μm)	Weight percent%	Cumulative wt%
<20	10	8	8
20–40	30	18	26
40–60	50	31	57
60–80	70	22	79
80–100	90	15	94
>100–120	110	6	100

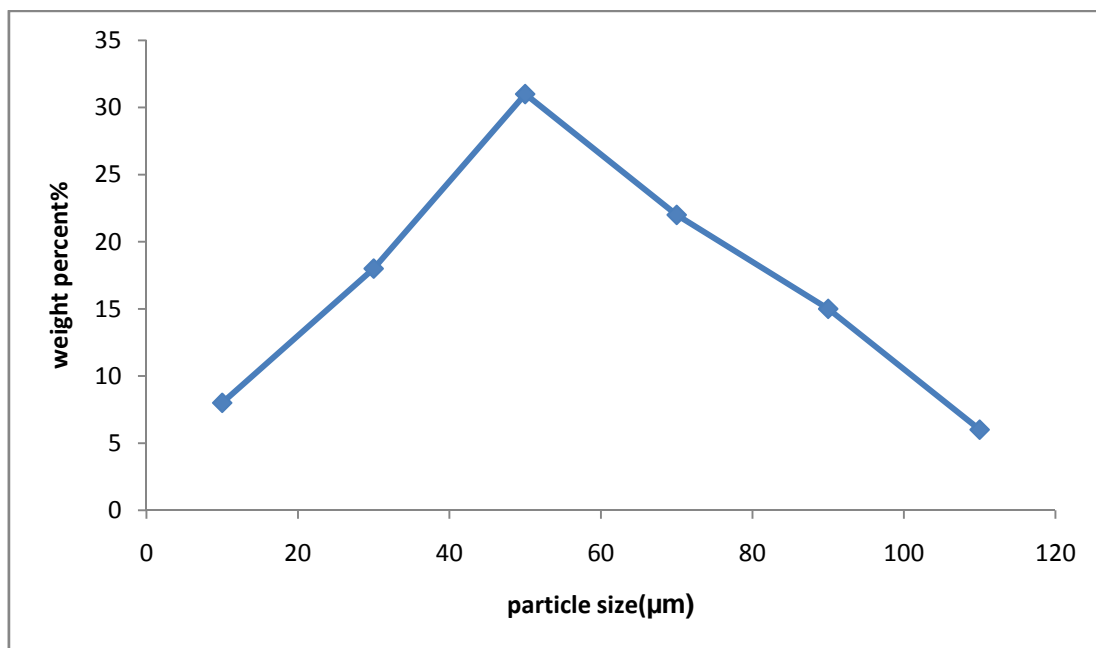


Figure (3.1) shows particle size distribution.

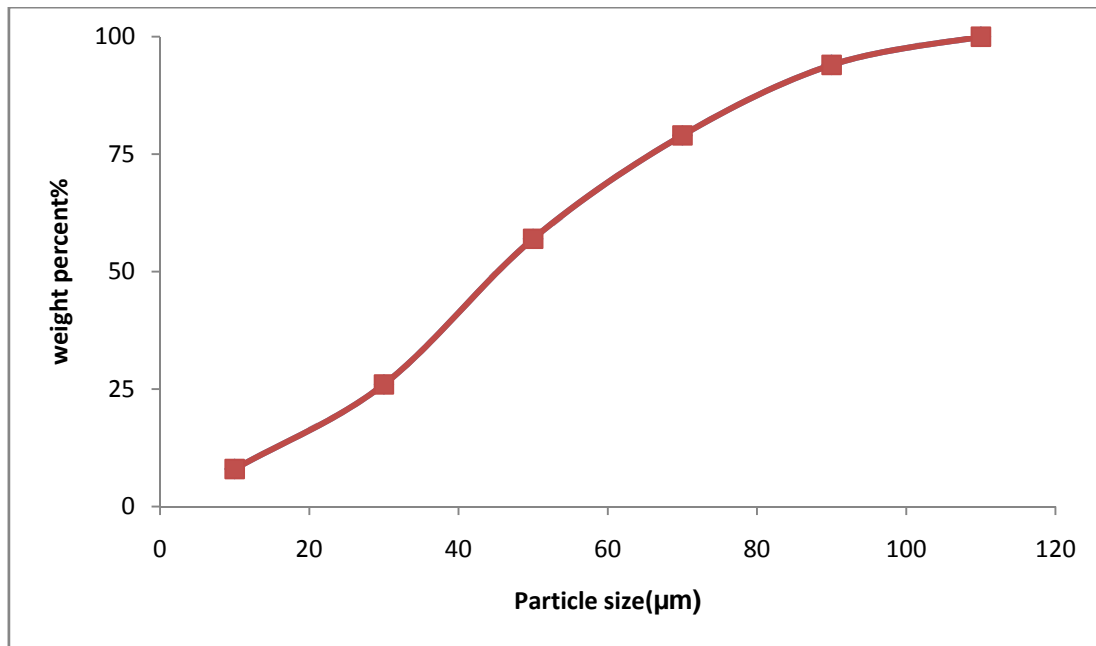


Figure (3.2) shows particle size analyses.

3.3 Effect of mould dimension on density of compacts

Two types of dies (10,15mm) in diameter were used to prepare specimens.

A specific amount of Al powder (2.12 gm) was used to prepare number of samples using different pressures (500, 1000, 1500, 2000, and 2500) psi .The volume and the density of samples were calculated (table 3.2). It was found that; using die with radius 10 mm, p (2500 psi) will gives good green density and good contact point between particles of Al powder.

Table (3.2) values of given pressure, and actual pressure for pure Al with mould dimension, 10, and 15mm.

	Die radius 10mm	Die radius 15mm
Given pressure(instrumental psi)	Actual pressure(psi/cm ²)	Actual pressure (psi/cm ²)
500	637	283
1000	1273	566
1500	1911	850
2000	2548	1132
2500	3183	1414

3.4 Effect of particle size on density and hardness of compacts

Characteristics of starting powder such as particle size, shape, aggregates, and degree of agglomeration have a great influence on densification. An ideal powder should have small particle size, non-agglomeration, and equiaxed particle shapes.

According to section (2.4.2) the powder were used for prepare samples had different particle size, Table (3.1). It was found that the powder have a large amount of particle size (40-60) μm .

Smaller particle size may provide more strengthening than larger ones, because the particle spacing between fines grained materials smaller than

coarse grained ones. Higher density was obtained from smaller particle size ($<20\mu\text{m}$), there is no significance change in density with particle size $<20, 30, 40 \mu\text{m}$. Density change about 2% as particle size changed from $<20 - 100\mu\text{m}$ as shown in table (3.3).

It could be noted that the hardness increased as the particle size decreased reaching its optimum value (32.85) HV with average particle size $10\mu\text{m}$ as shown in Figure (3.3).

Table (3.3) values of particle size and relative density of pure Al heat treated at 600°C for 2 hr soaking time.

particle size	Relative density
<20	92.89
30	92.6
40	92
50	91.07
80	90.67
>100	90

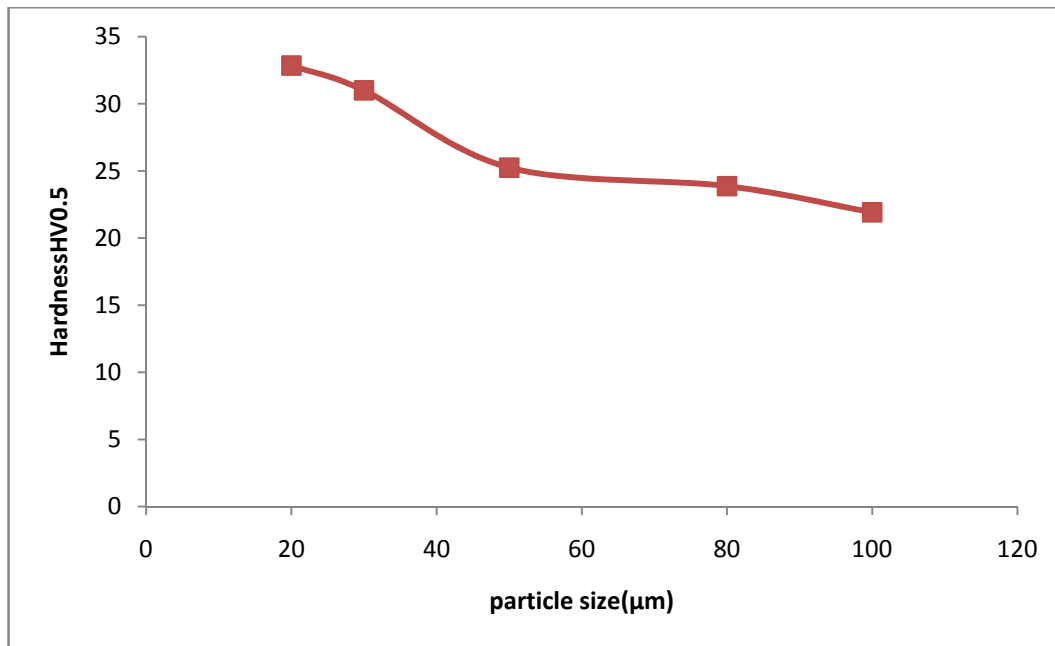


Figure (3.3) Hardness of pure Al sintered at 600°C for 2 hr soaking time with different particle size.

3.5 Effect of compaction pressure on density of compacts

Figure (3.4) shows the compressibility curve of pure Al powder, highest green density was achieved at the highest compaction pressure 2500 psi, after sintering process, specimens suffered about 2.1% volume shrinkage and the density approaches 92% of the theoretical density. In this experiment the die radius is equal to 10mm.

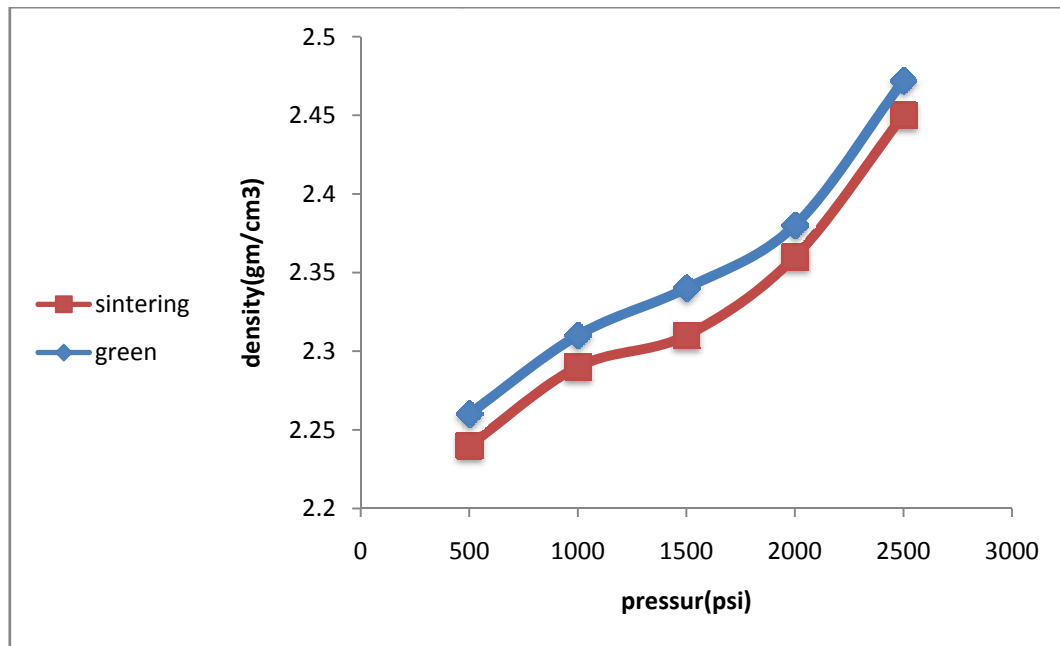


Figure (3.4) Relative green density and sintering density of pure Al sintered at 600°C for 2 hr soaking time as a function of compaction pressure.

3.6 Effect of boron carbide adding on density of compacts

Relative density has been calculated by dividing true density by calculated density which is calculated using equation (2.3).

Calculated density for

- Al+5%B₄C is 2.68
- Al+10%B₄C is 2.669

The density of MMCs decreased from 2.45 g/cm³ to 2.17 g/cm³ as a minimum with 10% boron carbide reinforcement. By considering two extremities of the materials such as pure Al and pure B₄C the density was expected to vary in-between 2.7 g/cm³ and 2.51gm/cm³. This in the case with the theoretical density which is difficult to get in powder metallurgy techniques. The observed density is with what is expected. Figure (3.5) shows the sintering density with B₄C and heat treated at 600°C for 2 hr soaking time.

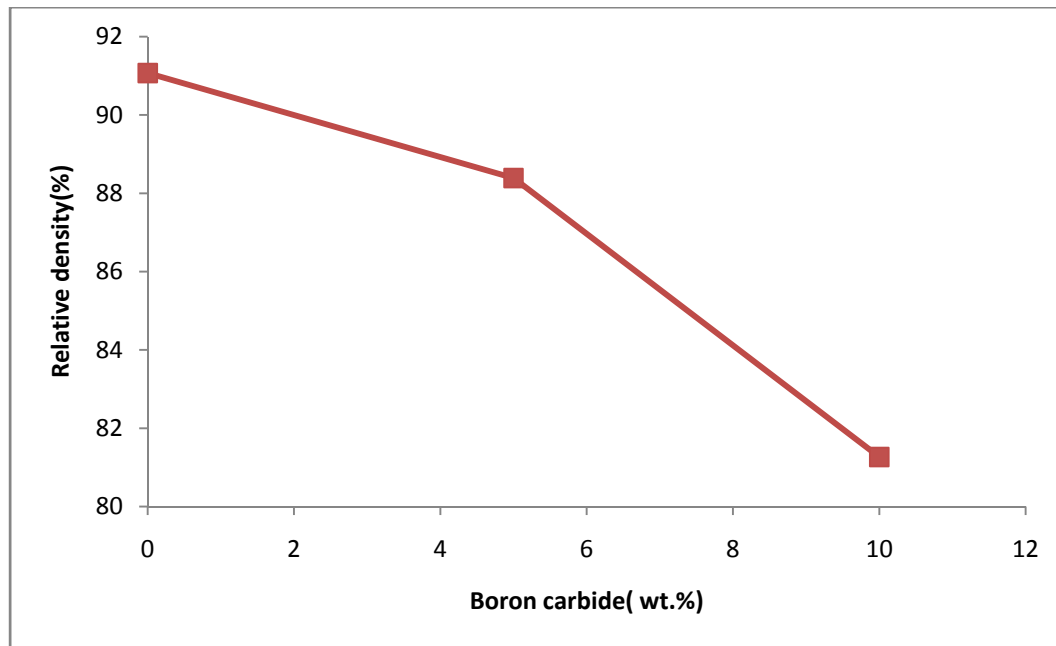


Figure (3.5) sintering density with different B₄C reinforcement percent % heat treated at 600°C for 2 hr soaking time.

3.7 Mechanical tests

3.7.1 Vickers Hardness Test

Vickers hardness test have been applied for:

- Pure Aluminium powder compacts with average particle size 60µm with pressure 500, 1000, 1500,2000,2500psi heat treated at 600°C
- Pure Aluminium compacts with average particles size <20, 30,40,60,80,>100 µm using 2500 psi pressure
- Pure Al, Al+5%B₄C, Al+10%B₄C with average particle size 60µm heat treated at temperatures 400,450,500,550,600,650°C.
- Pure Al, Al+5%B₄C, and Al+10%B₄C heat treated at soaking times 2,4,6,8,10h.

These tests have been carried out at room temperature. The results are the mean values based on three tests. From these tests Vickers hardness was obtained straightly. Figure (3.6) to (3.9) illustrates the hardness

change with pressure, temperature, soaking time and weight percent of B_4C .

Hardness increased after adding boron carbide, the B_4C particles increased the hardness over the entire temperatures interval that was analysed. Material presented high hardness values due to the homogenous distribution of the B_4C particles, enhancing the hardness of the unreinforced aluminum (27.7HV) to 57.87HV, 94.89HV for 5% and 10% respectively at optimum temperature used ($650^\circ C$). The hardness increased in aluminum based boron carbide composites with increasing reinforcement weight percent, hardness of Al+10% B_4C wt% is greater than Al+5% B_4C wt% as shown in figure (3.7) which also shows effect of temperature on hardness. It was intended to comprise the nearest range of sintering temperature under the melting temperature of aluminum ($660^\circ C$) without reaching it, best result was obtained at $T=650^\circ C$ for 5% B_4C , 10% B_4C . Hardness of Al+10% B_4C heat treated for 2h is greater than Al+5% B_4C heat treated at the same time by 38%. The hardness slightly changed at temperature range $400-550^\circ C$ and began to change sharply in temperature range $550-650^\circ C$ as shown in figure (3.7).

The hardness of various B_4C % composites and the pure Al are plotted in figure (3.9). Test results shows an increase in hardness with an increase in reinforcement (B_4C) content in the aluminum. The hardness of 5% B_4C heat treated at $650^\circ C$ is approximately twice the hardness of pure aluminum heat treated at the same temperatures.

The hardness of 10% B_4C heat treated at $650^\circ C$ is 2.5 of the unreinforced as shown in figure (3.9). hardness of Al+5% B_4C , and Al+10% B_4C heat treated at $600^\circ C$ for 10 hr changes 61.62% and 99.39% respectively as shown in figure(3.8).

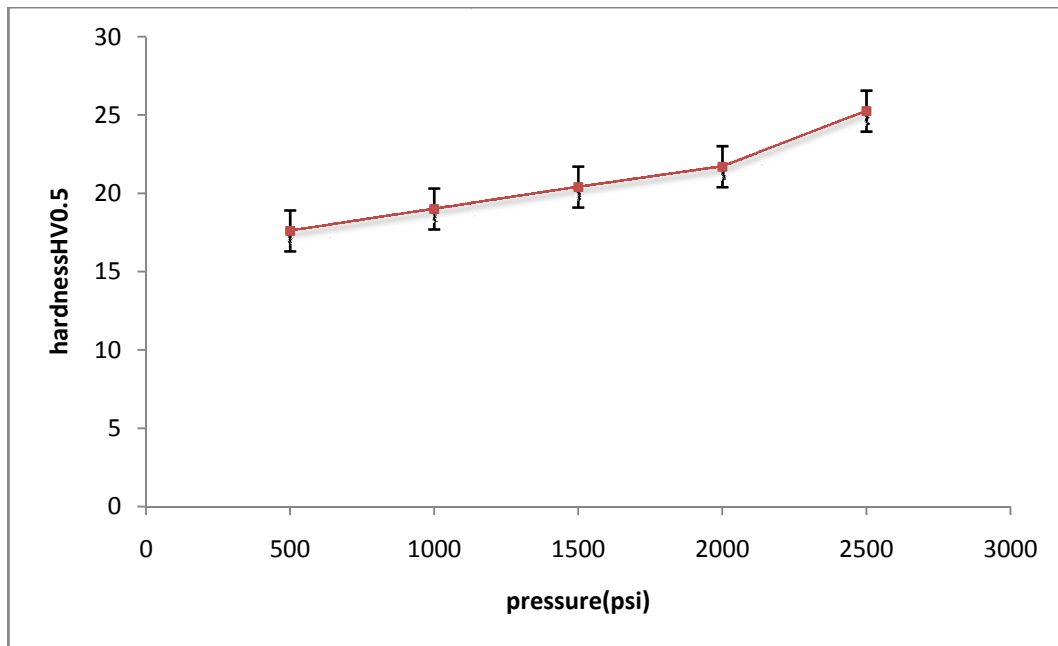


Figure (3.6) Hardness of pure Al sintered at $T=600^{\circ}\text{C}$ for 2hr soaking time compacted at different pressures.

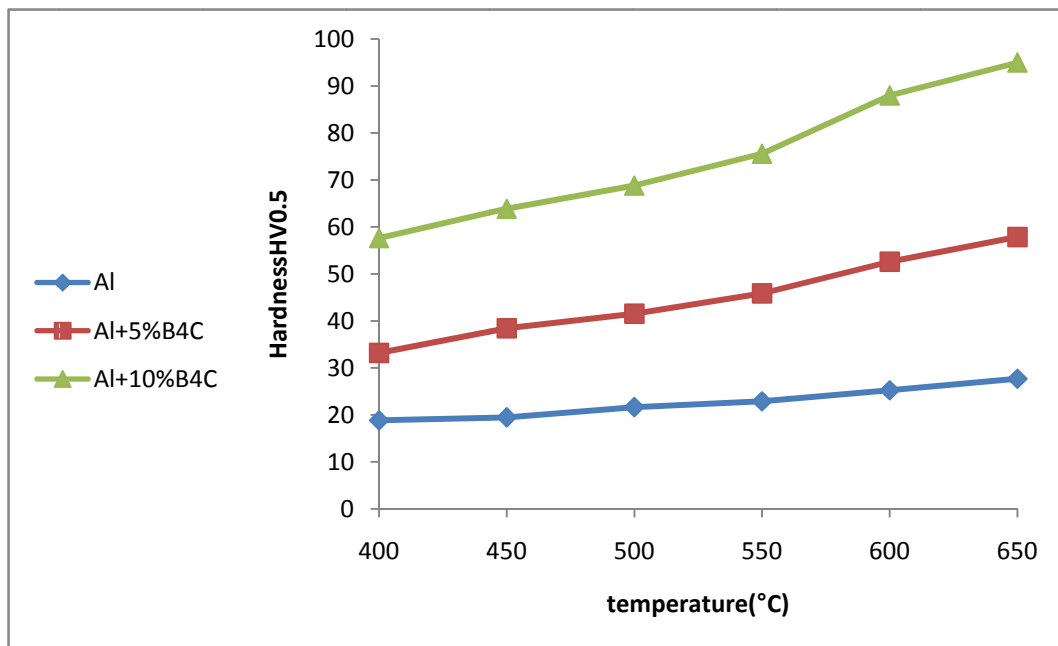


Figure (3.7) Hardness of pure Al, 5%, 10%reinforced B_4C heat treated at different temperatures.

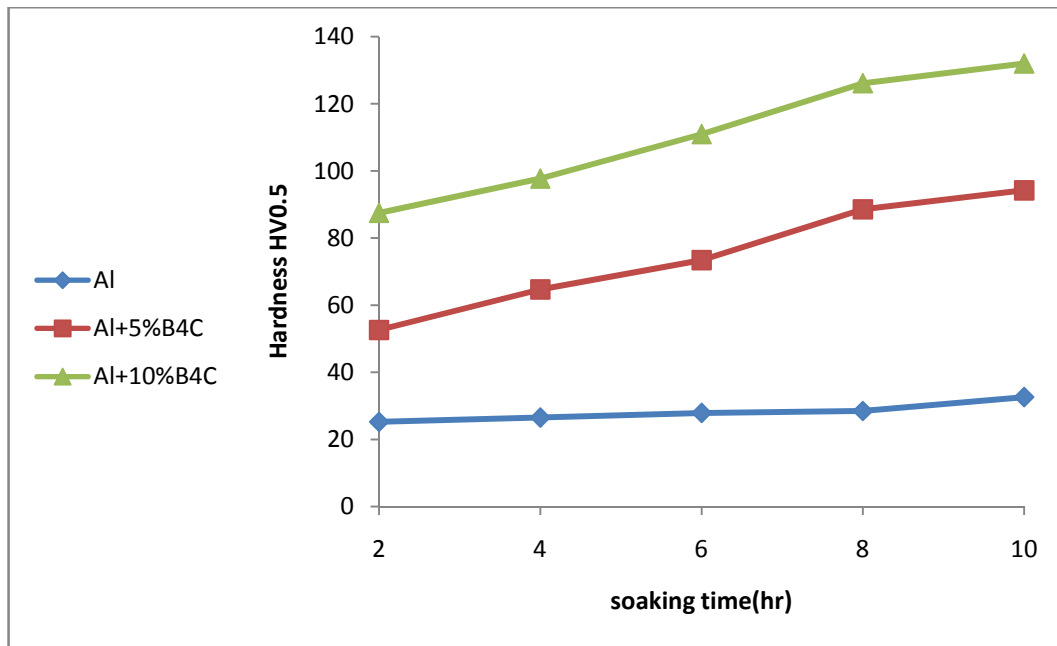


Figure (3.8) Hardness of Al, 5%, 10%B4C sintered at 600°C with different soaking times.

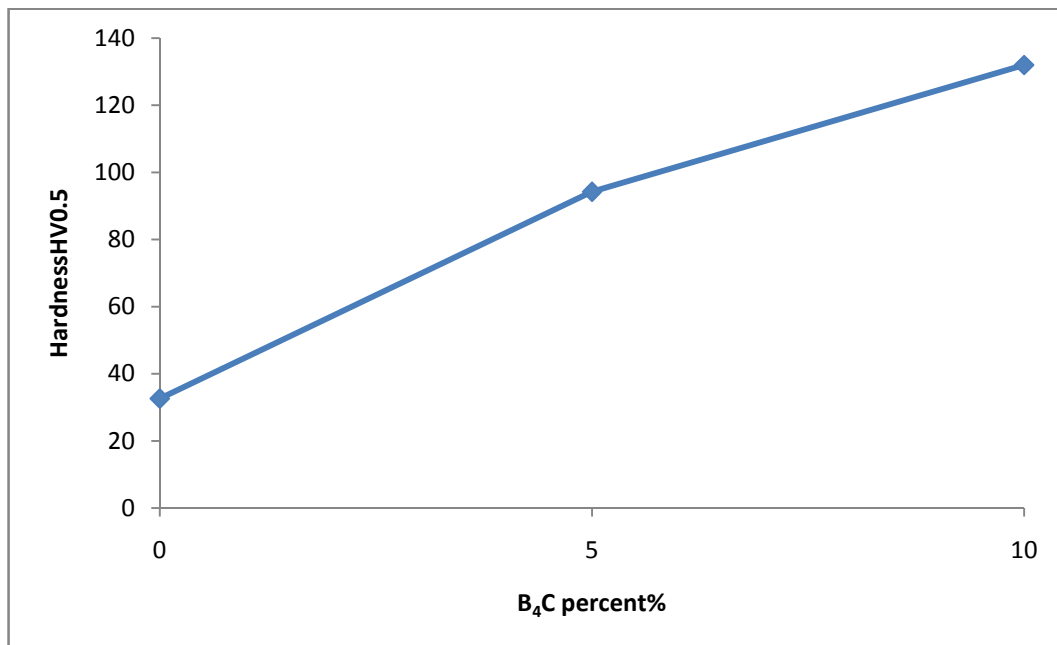


Figure (3.9) Effect of reinforcement percent on hardness of Al and Al with B₄C composites heat treated at 600°C for 10hr soaking time.

3.7.2 Compression test:

Compression testing is one of the most used methods for determining the modulus of elasticity and compression yield stress of a material.

Compression test have been applied for:

- Pure Al, Al+5%B₄C , Al+10%B₄C with average particle size 50µm heat treated at temperatures 400,450,500,550,600,650°C.
- Pure Al, Al+5%B₄C, and Al+10%B₄C heat treated at soaking times 2,4,6,8,10h.

The yield strength values were substantially influenced by the addition of B₄C particles as shown in figure (3.10). The compressive strength increased as the reinforcement increase. The yield strength ranged from 73.88 to 119.3MPa and from 91.63 to 153MPa for 5%, and 10% weight percent B₄C over the entire range of temperature as shown in figure (3.10).there is no significant change in compressive strength as the temperature change from 400–550°C. The compressive strength increased from 11 to 35.23% for 5wt% and from 23.64 to 38.36% for 10wt% as temperature change from 550–650°C wt% respectively as shown in figure (3.10).the ultimate strength change 47%,139% for 5%B₄C, and 10%B₄Crespectively this is compared with pure aluminium as shown in figure (3.12).

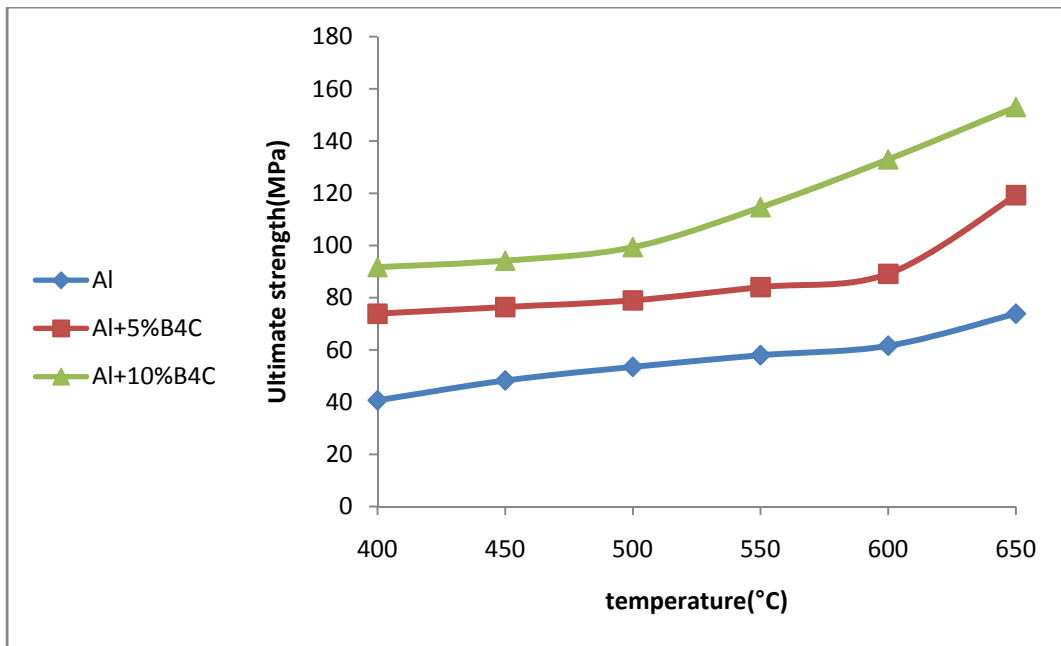


Figure (3.10) compressive strength of Al, 5%B₄C, 10% B₄C heat treated at different temperatures.

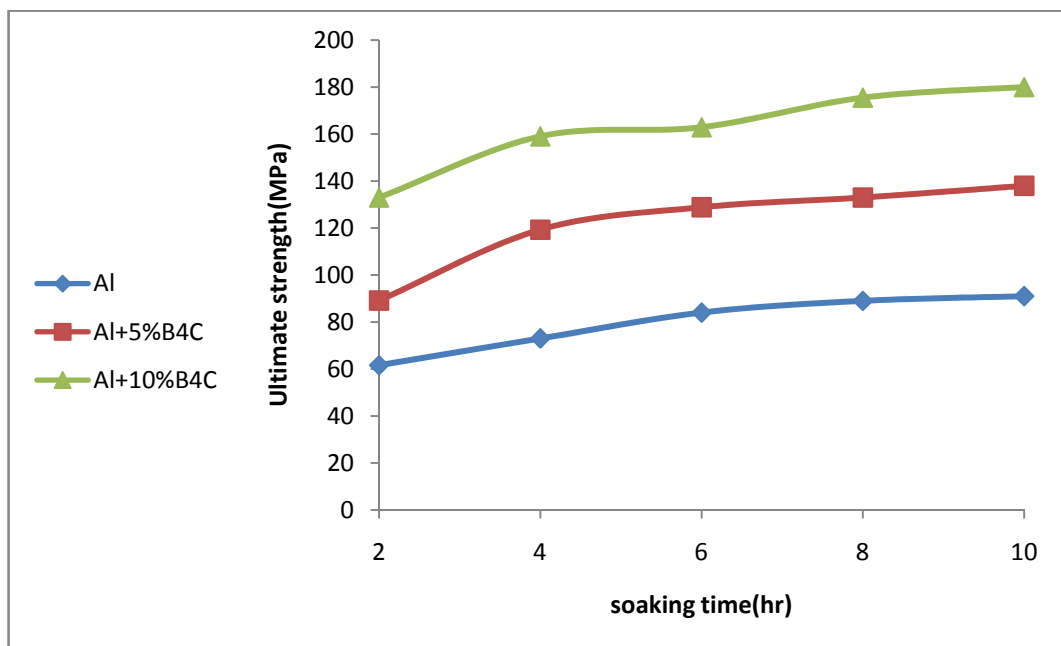


Figure (3.11) compressive strength of Al, 5%B₄C, 10%B₄C sintered at 600°C different soaking time.

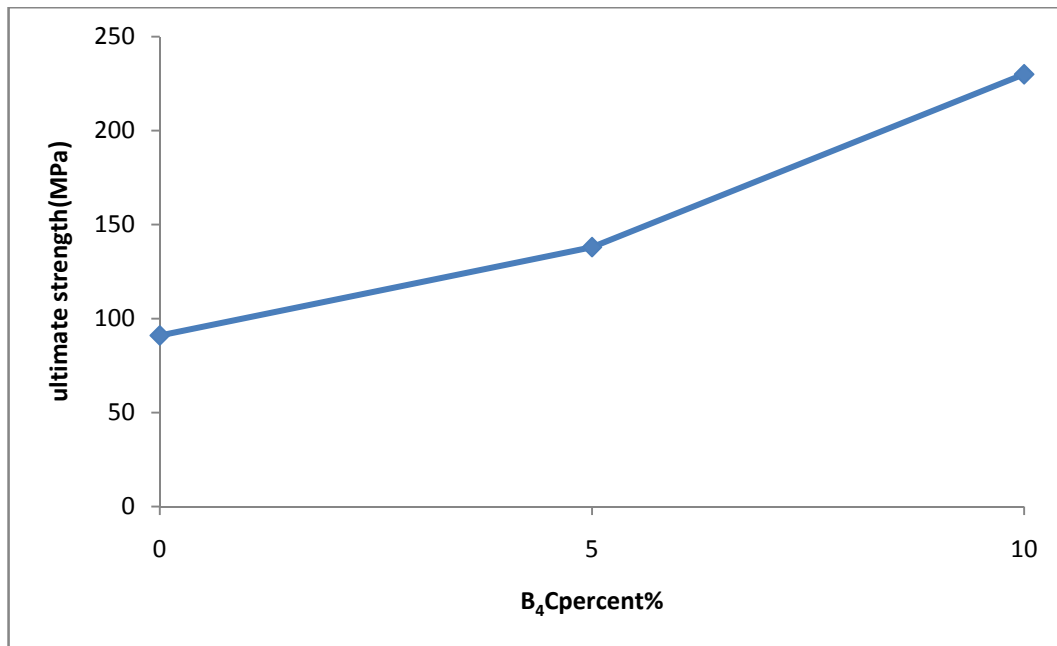


Figure (3.12) Effect of reinforcement percent on ultimate strength of Al and Al with B₄C composites heat treated at 600°C for 10 hr.

3.7.3 Young's Modulus

The modulus of elasticity has been calculated from the slope of stress-strain plotted in figure (3.15), (3.16). Test result shows an increase in elastic modulus with an increase in B₄C percent. The elastic modulus of the 5% B₄C heat treated at 650°C is 1.94, which is 84.12% higher than that of pure aluminium heat treated at the same conditions. The elastic modulus of 10% B₄C heat treated at 650°C is 2.33, which is 123% higher than the modulus of elasticity of pure aluminium heat treated at the same conditions. The elastic modulus change 9%, 47%, 50% as the soaking time change from 2 to 10 hr for Al, 5% B₄C, 10% B₄C respectively.

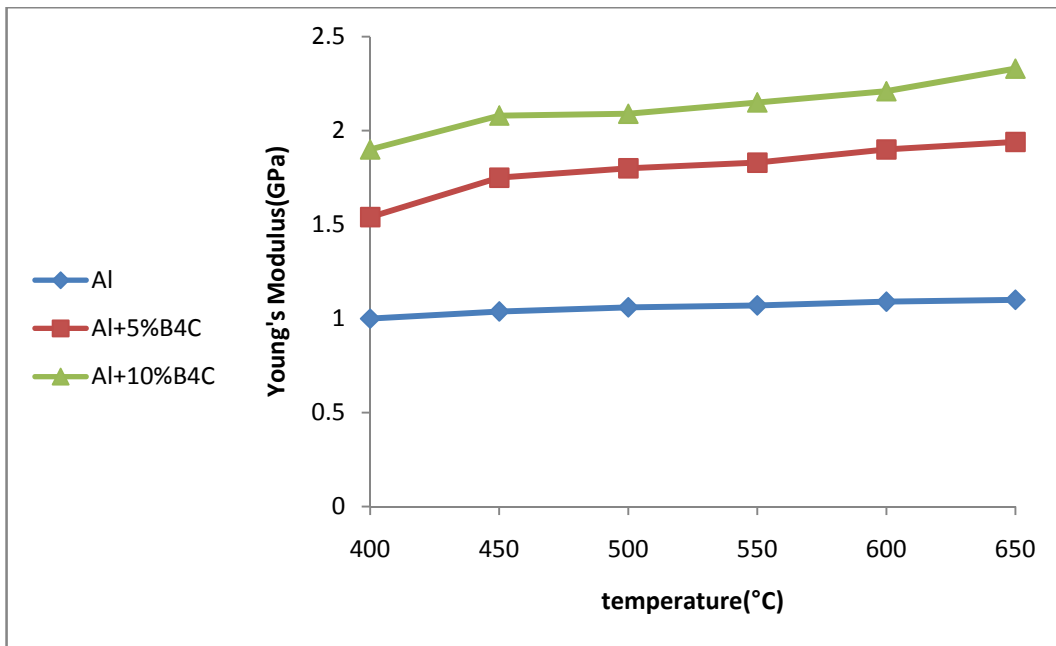


Figure (3.13) Modulus of elasticity versus temperature.

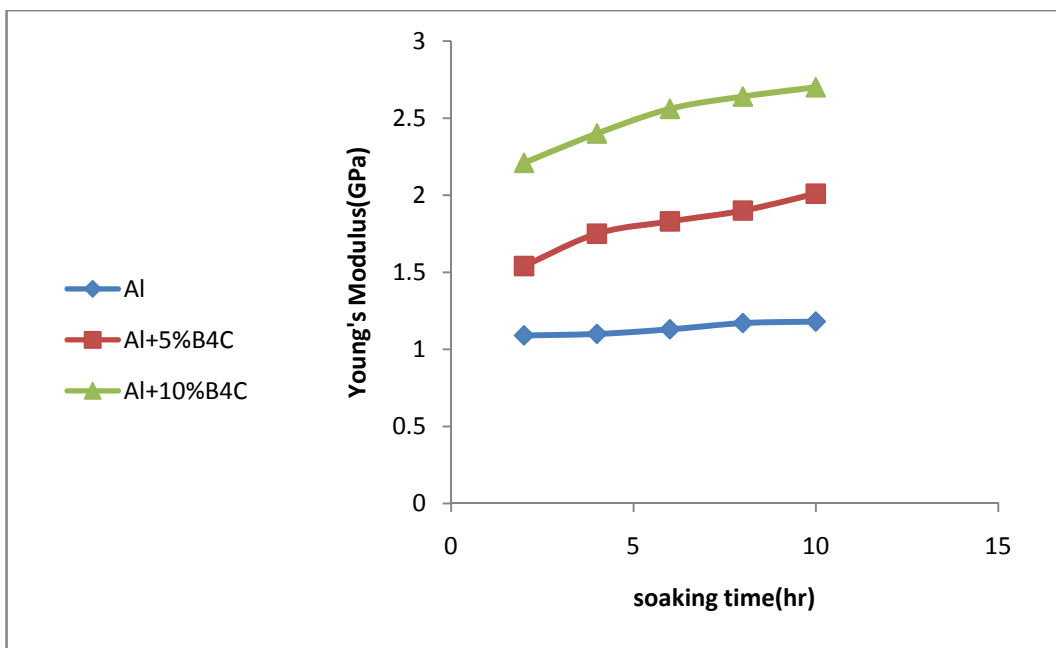


Figure (3.14) Modulus of elasticity versus soaking time.

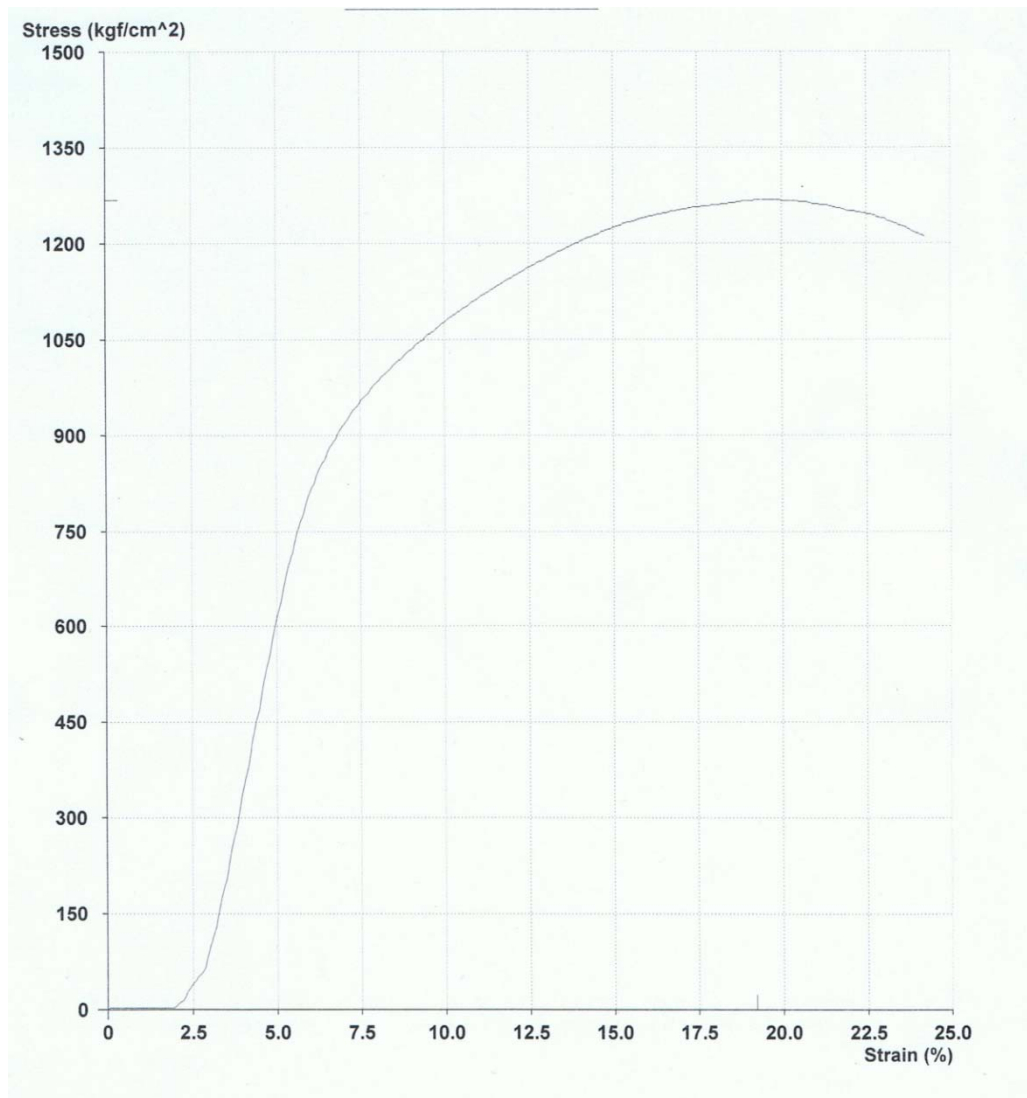


Figure (3.15) stress-strain curve for 5%B4C heat treated for 10 h.

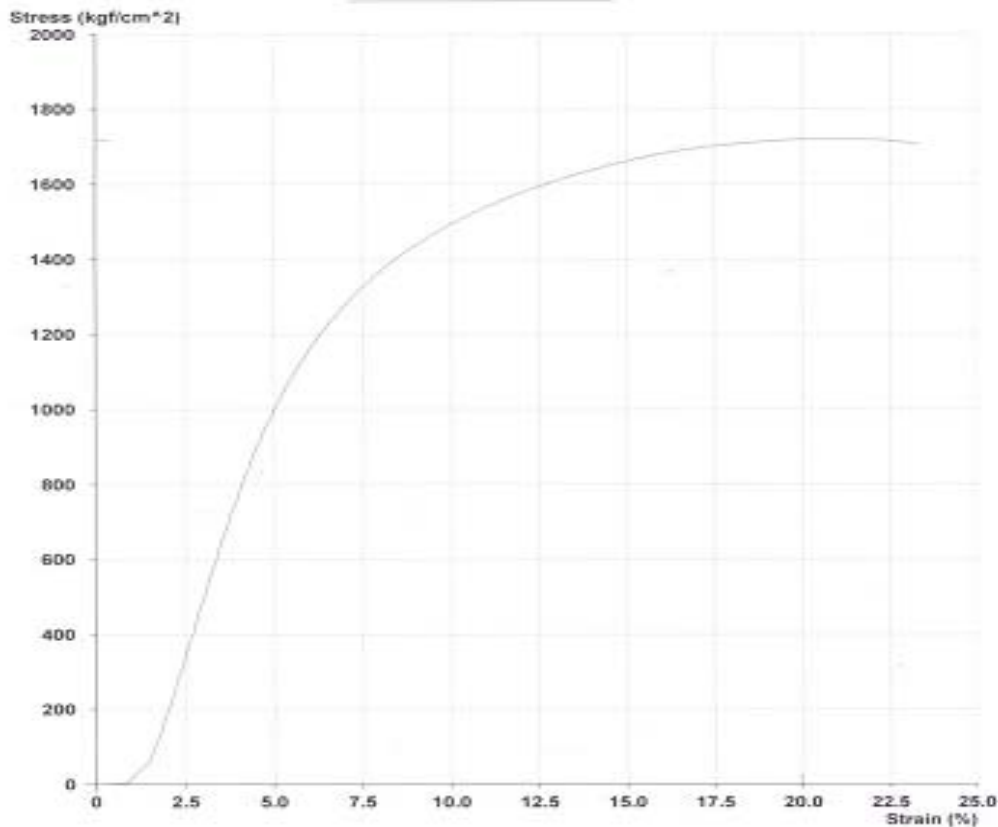


Figure (3.16) stress-strain curve for 10%B₄C heat treated for 10 hr.

3.8 Irradiation effect on hardness

Boron carbide is extensively used as a control rod material, because of its superior properties such as great neutron absorption capacity, high melting temperature, and light weight. The helium produced by $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction is released from the samples, the retained helium in the pellets caused the samples swelling, because of that the hardness of 5%,10%B₄C decreased. The main factor effect on swelling is accumulation of helium produced by $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction in samples contain 5,10% B₄C. The hardness of aluminium was increased after irradiation this is shown in figure (3.17).This increased is observed when pure metal is subject to neutron irradiation. The hardness of Al+5%B₄C and Al+10%B₄C decreased after irradiation,its also noticed that such

effect is more pronounced in 10% wt B_4C compared with 5% B_4C this is shown in figure (3.18) and (3.19).

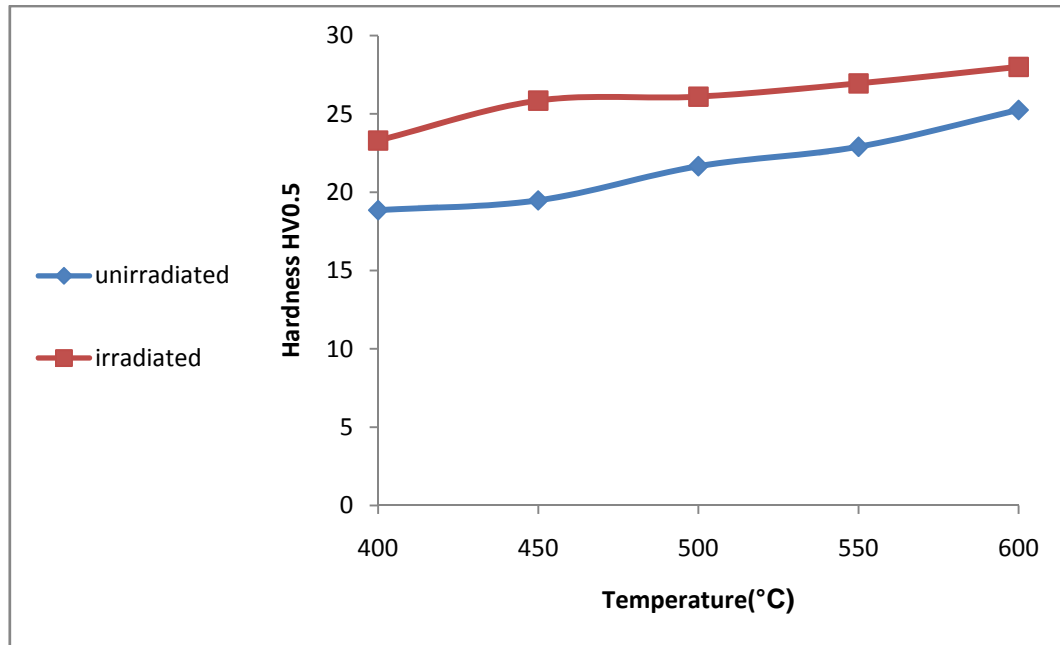


Figure (3.17) Irradiation effect on hardness for pure aluminium heat treated for 2hr soaking time.

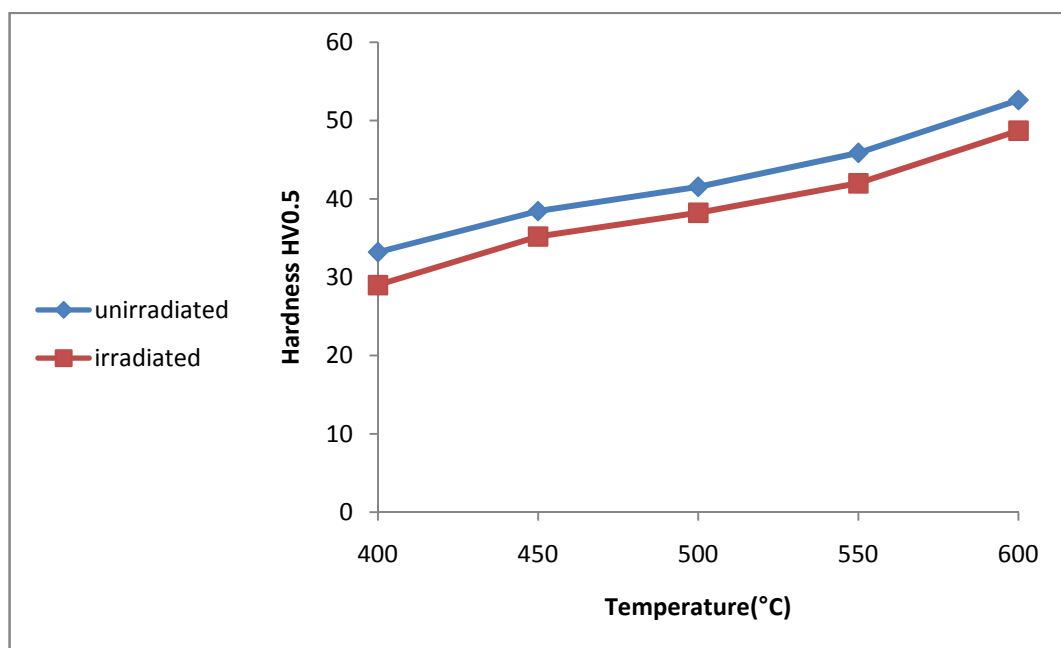


Figure (3.18) Irradiation effect on hardness for Al+5% B_4C heat treated at 600°C for 2 hr soaking time.

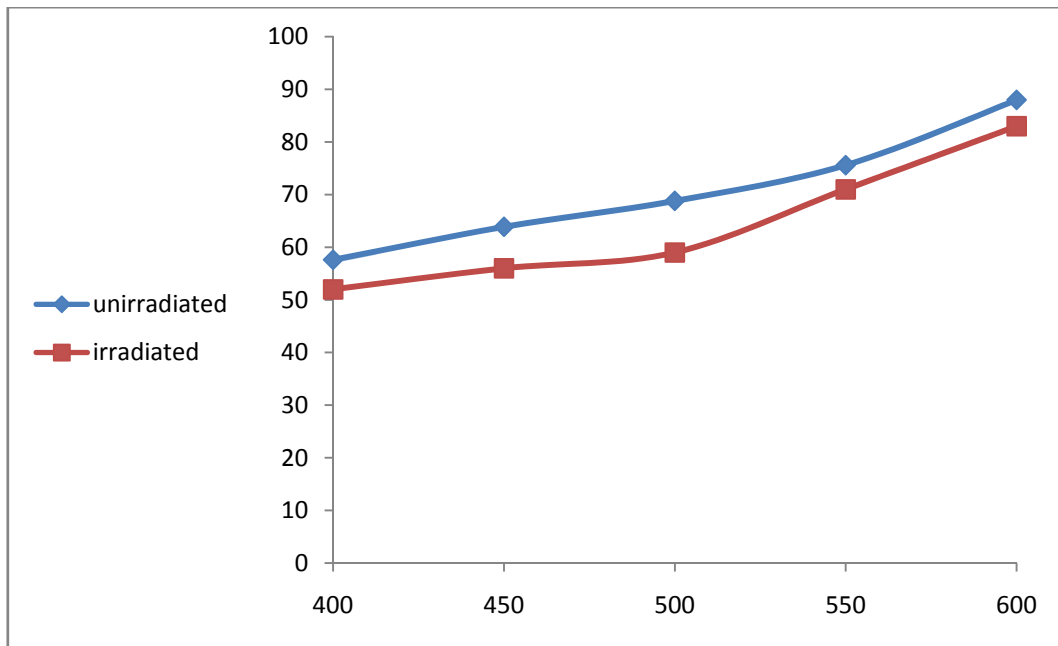


Figure (3.19) Irradiation effect on hardness for Al+10%B₄C heat treated at 600°C for 2hr soaking time.

Chapter Four

Conclusions and Recommendations

4.1 Conclusions

The main important conclusions that can be drawn from this work are as follows:

1. It was found that good green density and good contact point between particles of Al powder were obtained by using die with radius 10 mm, pressure (2500 psi).
2. Hardness increased approximately 2.5 fold as B₄C percentage increased from 0 to 10% B₄C.
3. There is no significant increased in hardness at temperature range 400–550°C, and change significantly at temperatures 600,650°C.
4. Compressive strength increased approximately 47%, 139% for 5%B₄C, and 10%B₄C respectively this is compared with pure Al.
5. Modulus of elasticity for 10%B₄C heat treated at 650°C increased 123% compared with pure Al.
6. The hardness of aluminium was increased after irradiation; The hardness of Al+5%B₄C and Al+10%B₄C decreased after irradiation, it's also noticed that such effect is more pronounced in 10% wt B₄C compared with 5%B₄C.

4.2 Recommendations

1. Use ball mill for better homogenisation of samples
2. Study bending strength for pure and reinforced aluminium
3. Study thermal conductivity for pure and reinforced aluminium
4. Study the effect of particle size on the mechanical properties of aluminium
5. Use higher temperature (approach 800°C) where near liquid phase sintering takes place
6. Study the wear and creep properties for pure Al and reinforced with B₄C.

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الخلاصة

لقد استهدف البحث دراسة تأثير المعاملة الحرارية على الخواص الميكانيكية للمترابك المنيوم-كاربيد البورون المصنعة بطريقة تكنولوجيا المساحيق. تم قياس قابلية الانضغاط لمسحوق الالمنيوم باستعمال ضغوط مختلفة حيث تم اختيار اعلى ضغط استخدم (2500psi). تم تحضير النماذج باضافة 5%، 10% من كاربيد البورون الى مسحوق الالمنيوم بالاضافة الى مسحوق الالمنيوم وتم كبس المادة بضغط 2500psi. عملية التليد تمت بدرجات حرارة مختلفة ($400, 450, 500, 550, 600, 650^{\circ}\text{C}$) وبزمن أنضاج (2, 4, 6, 8; 10 hr). الفحوصات الميكانيكية اظهرت زيادة نسبة الصلادة، الانضغاطية ومعامل المرونة بنسبة قليلة في درجات الحرارة ($400, 450, 500, 550, 600, 650^{\circ}\text{C}$) وزيادة بنسبة اكبر بدرجات الحرارة ($600, 650^{\circ}\text{C}$).



جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة النهرين
كلية العلوم

**تأثير المعاملة الحرارية على بعض الخواص الميكانيكية
للمتراكب المنيوم-كاربيد البورون**

رسالة مقدمة الى
كلية العلوم - جامعة النهرين
وهي جزء من متطلبات نيل درجة ماجستير علوم في الفيزياء
من قبل

لمى جمال عباس الرماحي
(بكالوريوس علوم في الفيزياء 2006 م)

بإشراف

رئيس الباحثين د. سعد رحمة الله

م. د. قاصد عبد الستار

تشرين الاول-2009

شوال-1430