JANUARY 2016

M.Sc. in Mechanical Engineering

MUHAMMED PAKSOY

UNIVERSITY OF GAZİANTEP

GRADUATE SCHOOL OF

NATURAL & APPLIED SCIENCES

EFFECT OF ASPECT RATIO AlB_2 FLAKES ON MECHANICAL AND PHYSICAL PROPERTIES OF ALUMINIUM BORON ALLOYS

M.Sc. THESIS

IN

MECHANICAL ENGINEERING

BY

MUHAMMED PAKSOY

JANUARY 2016

Effect of Aspect Ratio AlB_2 Flakes on Mechanical and Physical Properties of Aluminium Boron Alloys

M.Sc. Thesis

in

Mechanical Engineering

University of Gaziantep

Supervisor

Assoc. Prof. Dr. Necip Fazıl YILMAZ

by

Muhammed PAKSOY

January 2016

REPUBLIC OF TURKEY
UNIVERSITY OF GAZİANTEP
GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES
DEPARTMENT OF MECHANICAL ENGINEERING

Name of the thesis: Effect of Aspect Ratio AlB_2 Flakes on Mechanical and Physical Properties of Aluminium Boron Alloys

Name of the student: Muhammed PAKSOY

Exam date: January, 2016

Approval of the Graduate School of Natural and Applied Sciences

Prof. Dr. Metin BEDİR
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science.

Prof. Dr. Mehmet Sait SÖYLEMEZ
Head of Department

This is to certify that we have read this thesis and that in our consensus/majority opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

Assoc. Prof. Dr. Necip Fazıl YILMAZ
Supervisor

Examining Committee Members:	Signature
Prof. Dr. Hüseyin FİLİZ	....
Prof. Dr. Ömer EYERCİOĞLU	....
Prof. Dr. Abdulkadir EKŞİ	....
Assoc. Prof. Dr. Ahmet ERKLİĞ	....
Assoc. Prof. Dr. Necip Fazıl YILMAZ	....

iii

I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Muhammed PAKSOY

ABSTRACT

EFFECT OF ASPECT RATIO AlB_2 FLAKES ON MECHANICAL AND PHYSICAL PROPERTIES OF ALUMINIUM BORON ALLOYS

PAKSOY, Muhammed

M.Sc. in Mechanical Engineering

Supervisor: Assoc. Prof. Dr. Necip Fazıl YILMAZ

January 2016

66 pages

In this study, cast Al-B alloys which have different aspect ratio (length/width) AlB_2 flakes were produced. The main objective of this investigation was to explore effect of aspect ratio AlB_2 flakes on the mechanical and physical properties of the cast Al-B alloys. Borax powders ($Na_2B_4O_7.10H_2O$) wt. 10% was added to molten aluminium at 1000°C which is the peritectic temperature on Al-B phase diagram by holding in the furnace during 45 minutes to fabricate AlB_2 compounds. The cast Al-B alloys were cooled in water, air and furnace. Heat treatment was applied to cast Al-B alloys by heating in the furnace at 550°C with different holding times 5h, 15h, 30h, 50h and 70h to acquire different aspect ratio particles. Microstructures of Al-B alloys were observed and aspect ratios of the flakes were measured by using microscope. All samples were subjected to mechanical and physical tests such as hardness, tensile, impact and electrical conductivity. As a result of heat treatment for 15 hours, an increase of 14.5% on hardness, an increase of 16% on tensile strength, an increase of 19% on impact energy and an increase on electrical conductivity were obtained in comparison with non-heat treated samples. Reinforced AlB_2 particles in aluminium matrices were also observed by using Scanning Electron Microscope.

Keywords: AlB_2 Flakes, Boron, Aspect Ratio, Al-B Alloy, Microstructure

ÖZET

ALÜMİNYUM BOR ALAŞIMLARININ MEKANİK VE FİZİKSEL ÖZELLİKLERİNE AlB_2 PARTİKÜLLERİ ASPEKT ORANLARININ ETKİSİ

PAKSOY, Muhammed
Yüksek Lisans Tezi, Makine Mühendisliği Bölümü
Tez Yöneticisi: Doç. Dr. Necip Fazıl YILMAZ
Ocak 2016
66 sayfa

Bu çalışmada, farklı aspekt oranlarına (boy/en) sahip AlB_2 parçacıklı Al-B alaşımları üretildi. Bu araştırmanın temel amacı, AlB_2 parçacıklarının aspekt oranlarının Al-B alaşımlarının mekanik ve fiziksel özellikleri üzerindeki etkisini incelemekti. AlB_2 bileşiği elde etmek için ağılıkça % 10 miktarında toz şeklindeki boraks kristalleri ($Na_2B_4O_7.10H_2O$), Al-B faz diyagramında peritektik sıcaklık olan 1000^oC'de eritilmiş alüminyum içerisine ilave edilerek fırın içerisinde 45 dakika boyunca bekletilmiştir. Elde edilen Al-B alaşımları suda, havada ve fırın içerisinde soğutuldu. Farklı aspekt oranlarına sahip parçacıklar elde etmek için 550^oC'de Al-B alaşımları fırın içerisinde sırasıyla 5, 15, 30, 50 ve 70 saat farklı sürelerde bekletilerek ısıl işlem uygulandı. Mikroskop kullanılarak Al-B alaşımlarının mikro yapıları gözlemlendi ve AlB_2 parçacıklarının aspekt oranları ölçüldü. Elde edilen alaşımlara çekme, sertlik, darbe gibi mekanik ve elektrik iletkenliği ölçümü gibi fiziksel testler uygulandı. 15 saat boyunca ısıl işlem uygulanan alaşımlarda, ısıl işlem uygulanmayanlara göre sertliklerinde %14.5, gerilme mukavemetlerinde %16, darbe enerjilerinde %19 ve elektrik iletkenliklerde artış elde edildi. Ayrıca alüminyum matris içerisine takviye edilen AlB_2 parçacıkları taramalı elektron mikroskobuyla incelendi.

Anahtar Kelimeler: AlB_2 Parçacıkları, Bor, Aspekt Oranı, Al-B Alaşımı, Mikroyapı

To My Parents Ayşe, Hasan and My Brother Abdullah

ACKNOWLEDGEMENT

I would like to express my deepest respect and most sincere gratitude to my supervisor, Assoc. Prof. Dr. Necip Fazıl YILMAZ, for his guidance and encouragement at all stages of my work. His constructive criticism and comments from the initial conception to the end of this work is highly appreciated.

I am very grateful to Musa YILMAZ, Emre SARIKAYA, Serhat MUSTAFA and Kemal GÜRPINAR for their support and hardworking throughout this study. I am also very grateful to Burak ŞAHİN for his helpful comments and continuous support and staff of Mechanical Engineering Department Workshop and Laboratories, especially Seyit BOSTANCI and Mehmet TAŞDEMİR for their help for preparation of test specimens.

I also would like to thank Prof. Dr. Metin BEDİR and Ziya GESOĞLU for their valuable comments and help throughout laboratory work for the determination of electrical conductivity of specimens.

I would also like to thank KOSGEB Gaziantep Service Center Manager Gökhan DOĞAN and my colleagues.

Also, I would like to thank my mother Ayşe, my father Hasan and my brother Abdullah for their continuous help, encouragement and for their understanding. It is to them that I dedicate this thesis.

Finally, I would like to serve my gratitude to examining committee members spending their valuable time for attending my M.Sc. qualification.

CONTENTS

Page

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

Modern technology era, rapid development of science and technology, modern production and manufacturing systems and methods lead to increasing the importance of new and improved materials and metal alloys, for the sake of fast and low-cost fabrication, low-weight, increased strength, high hardness, resistance to wear and corrosion, easy machining and ability to absorb shocks.

Metal alloys play a significant role due to their enhancing mechanical and physical properties by dispersing each other and incorporation of two or more than different materials within atomic bonding. Especially, aluminium alloys are more popular among the other metal alloys due to their superior physical and mechanical properties [1].

Aluminium is a light metal which have many important properties such as high strength, low density-low weight, superior malleability and formability, excellent weldability, easy machining, perfect corrosion resistance, high thermal and electrical conductivity. Aluminium alloys are mainly preferred in automobile and aerospace industry due to their high strength, low-weight ratio, excellent corrosion resistance and good machinability. Structural components produced by aluminium, have wide application field in daily life from automotive to aerospace, medical to military industry and from transportation to the construction industry [2].

Aluminium alloys have been widely investigated during recent years. Aluminium is generally alloyed with copper, manganese, silicon, magnesium, zinc and boron. Aluminium alloys also have a large variety of mechanical properties depending on type and amount of alloying elements [3].

Turkey has 73% of Boron reserves in all over the world. Rich and strategically important Boron reserves must absolutely be converted to products to increase added-value of the Boron products. All of physical and mechanical properties of the Boron and compounds must be known. In order to convert Boron to products, characterization of boron must be investigated. Methods and processes must be discovered how to improve physical and mechanical properties of boron. Although Boron reserves takes an important place, efforts for investigation of characterization and features are not enough to produce finished product. Some of the universities and research centres attempt to search different properties of Boron.

Ozturk and Yilmaz [4] studied on improvement of mechanical properties of AA1070 aluminium by the addition of borax under different holding conditions. In their study, different amount of borax was dissolved in pure commercial aluminium to characterize different Al-B alloys by considering different amount of borax, holding time and holding temperature. In this study, producing master alloys and examining their mechanical properties were consisted of the important step for this progressive study.

In this thesis, Borax powders ($Na_2B_4O_7.10H_2O$) wt. 10% was added to molten aluminium at 1000°C which is the peritectic temperature on Al-B phase diagram to fabricate AlB_2 compounds. The effect of aspect ratio on mechanical and physical properties of boron particles strengthened in aluminium metal matrix was investigated. The cast Al-B alloys were cooled in different cooling conditions such as water, air and furnace. Heat treatment process was applied to cast Al-B alloys by heating in the furnace at 550 °C to obtain different aspect ratio particles during different holding times 5h, 15h, 30h, 50h and 70h, respectively.

The targeted aim of this investigation was to explore the effect of aspect ratio on the mechanical and physical properties of the cast Al-B alloys. Aspect ratio is length to width ratio of the AlB_2 particles in the alloy. The cast Al-B alloys which have different aspect ratio were successfully produced. The microstructures of the Al-B alloys were observed by using microscope to measure aspect ratio of the samples. Scanning electron microscope (SEM) was used to depict the dissolved AlB_2 flakes in aluminium matrices.

All samples were subjected to mechanical and physical tests such as hardness, tensile, impact and electrical conductivity test.

In this study, following procedure was executed:

- An experimental set-up was constructed.
- Set of specimens were fabricated.
- Specimens were subjected to different heating and cooling conditions.
- Tensile strength, hardness and impact energy tests were conducted.
- Electrical conductivity of samples was measured.

In Chapter 2, a comprehensive Literature Review were presented based on previous works related to fabrication method of Al-B alloys, effective parameters on properties of Al-B alloys, characterization AlB_2 flakes and Al-B phase diagram and effect on boride particles formation.

In Chapter 3, general view on aluminium, boron, metal matrix composites and properties were shown. Boron reserves and its importance in Turkey were submitted.

In Chapter 4, experimental set up, test devices used in measurement of mechanical and physical properties, test standards for specimen preparation and metallographic works were demonstrated.

In Chapter 5, experimental results, graphs versus variable parameters and microstructural pictures were introduced. And also comparison of test results depended on change of parameters were indicated.

In Chapter 6, achievements, results and improvements of mechanical and physical properties of Al-B alloys throughout the study were explained and concluded.

Finally, recommendations for future studies were stated.

CHAPTER 2

LITERATURE SURVEY

2.1 INTRODUCTION

In literature survey chapter, related previous studies on Al-B alloys were presented. Studies such as production method of Al-B alloys, effect of heat treatment on mechanical and physical properties of Al-B alloys and types of boron sources in aluminium matrices were mentioned.

2.2 LITERATURE REVIEW ON ALUMINIUM BORON ALLOYS

Metal matrix composites (MMCs) are fabricated by means of unification of two or more different materials, which are form of reinforced particles or continuous-discontinuous fibres by dissolving and dispersion into a molten metal or partially solidified metals as a new material. Manufacturing methods to produce MMCs are mainly diffusion bonding, powder metallurgy, casting, pressure infiltration and spray codeposition [5].

A number of techniques which are called in-situ and ex-situ methods were performed to spread homogeneously reinforced particles in the metal matrix. These have advantages and also restrictions such as non-uniform dispersion of reinforced particles, micro porosity, control of matrix-reinforcement interface for manufacturing cost and applications. In situ method reinforced particles are yielded by chemical reactions and transformation of solid solution phase. In situ method is more practical and lower-cost than ex situ method because of good wettability, homogenous dispersion of particles and thermo mechanical properties [6].

Metal casting process is a low-cost production and practical method to produce metal alloys as well as complex shaped and big sized parts can be easily produced. However, some points play a restrictive role with the inclusion of the homogenous distribution of reinforced particles in the alloy, porosity level on the surface due to high temperature effect and gases ensuring wettability between alloyed materials and optimised chemical reaction [7].

Aluminium matrix composites are superior to non-aluminium metal matrix composites owing to their specific modulus, strength, low density, hardness, stiffness, excellent wear resistance, low-heat expansion coefficient, ease of fabrication and low manufacturing cost in engineering applications and industry [8].

Economically, Al-B alloys are broadly utilized to increase electrical conductivity of the aluminium alloys by eliminating transition metal impurities (such as titanium, vanadium, chromium and zirconium) which decrease conductivity of aluminium alloys in the manufacturing process. Boron is generally consumed by transition metal impurities in aluminium alloys and boron and transition metal impurities are precipitated through chemical reactions. Beside, Al-B alloys employ as a grain refiner in aluminium casting [9].

The first production study of AlB_2 particles were put into practice by Funk. Aluminium and boron were heated at 1000^oC [10].

In different pathways, production methods are carried out to fabricate Al-B alloys, with the inclusion of blending and reaction of borax powder ($Na_2B_4O_7.10H_2O$), fluoride salt (KBF_4), boron oxide (B_2O_3) in molten aluminium, mechanical alloying and electrolysis. Utilization of elemental boron as a boron source is a very expensive method to obtain Al-B alloys [11].

Hence, borax powders ($Na_2B_4O_7.10H_2O$) are practically and economically used as a boron source. Boron is reduced by melting aluminium during an in–situ exothermic reaction which is $Al_{(L)}+AlB_{12} \leftrightarrow AlB_2$ at peritectic temperature 973^oC in aluminium boron phase diagram. Borax powders are dissolved and dispersed in molten aluminium, yielding aluminium borides respectively, AlB_2 which is hexagonal and

AlB_{12} which is a tetragonal crystal structure. According to Al-B phase diagram, AlB_{12} is shown at the temperatures that are above peritectic temperature of 973^{o}C. AlB_2 crystals is shaped without higher borides such as AlB_{12} which is thought to confine strength of the Al-B alloys when reaction temperature passes over peritectic temperature and then melting is suddenly cooled to room temperature., AlB_{12} crystals transform to AlB_2 flakes [12].

AlB_2 crystals are characterized as low aspect ratio flakes and high aspect ratio flakes, which are both consistent below peritectic temperature. The transformation of low aspect ratio AlB_2 to high aspect ratio AlB_2 is achieved by execution of heat treatments, aging quenching operations to low aspect ratio AlB_2 flakes [13].

A research on the formation of AlB_2 in an Al-B master alloy was performed by Wang [14-15]. The formation of Al-3 wt. % B master alloy was fabricated via chemical reaction between KBF_4 and molten aluminium at 850^{o}C. Immersion method and vortex methods were applied to produce Al-B alloys by mixing KBF_4 to aluminium. Aluminium boride compounds were formed by dispersing of boron in the melted aluminium. Scanning electron microscope (SEM) displayed two types of aluminium boride particles. X-ray diffraction (XRD) analysis identified the particles as AlB_2 and higher boride AlB_{12}. The presence of AlB_{12} forms in AlB_2 is related to peritectic reaction of $Al_{(L)}+AlB_{12} \leftrightarrow AlB_2$. As a result of immersion method, AlB_{12} phase and agglomeration of boride particles were observed. However, the alloys obtained by using vortex method had uniform boride particle distribution.

A study of characterization of in-situ AA 6060 / AlB_2 metal matrix composite was investigated by Dragut at al [16]. An aluminothermic reaction between AA 6060 aluminium and KBF_4 at 850^{o}C was accomplished for boron reinforced aluminium alloys. Na_3AlF_6 (cryolite) was added as an activator to reduce energy level for the reaction. After 60 minutes holding time in the furnace, melting alloy was casted into metal mold. X-ray diffraction (XRD) and Scanning electron microscope (SEM) observations were approved the formation of AlB_2.

Production of Al-B alloy by heating Al/ KBF_4 powder blend was performed by Birol [17]. Powder metallurgy method was preferred to search the effect of thermal exposure of Al/ KBF_4 blend in the Al-B alloy fabrication. When temperature reached to minimum 490^{o}C, which is lower than aluminium melting point, the reaction between Al and KBF_4 initiated. As a result of the reaction, AlB_2 and $KAlF_4$ compounds formed. In the experimental part of this study, 5 g aluminium powder and 1.8 g KBF_4 salt were blended during 5 minutes and Al/KBF_4 mixture was ball-milled during 30 minutes by using hardened steel balls in the second part of the experiment. Different samples taken from Al/ KBF_4 mixture were heated up to 750^{o}C in differential scanning calorimeter (DSC) to seem response to thermal exposure. Another critical point in this study, unwanted AlB_{12} particles were not formed in the alloy during ball-milled process.

Birol [18] was also interested in response to thermal exposure of balled-milled aluminium –borax powder blends. Another boron source borax salt ($Na_2B_4O_7.5H_2O$) and aluminium were ball-milled and heated higher than 600^{o}C to produce Al-B alloys. Depending on descending of temperature, hexagonal-shaped AlB_2 particles were extended along Al-B alloy.1 hour is enough to executing a thermite reaction. In this research, 5 g of (3.67g Al and 1.33g $Na_2B_4O_7.5H_2O$) blend was prepared. In order to investigate the response to thermal exposure of the ball-milled, Al/$Na_2B_4O_7.5H_2O$ blend was heated to 1000^{o}C in differential scanning calorimeter (DSC). And then, heat treatment was applied to different blended samples during 1 hour at 600^{o}C, 700^{o}C and 800^{o}C, respectively. At the end of the study, thermal exposure of balled-milled aluminium –borax powder blends makes easy the thermite reaction was pointed out.

Afterwards, Birol [19] worked on aluminothermic reduction of boron oxide for the manufacture of Al-B alloys. Al and B_2O_3 particles were ball-milled and then heated over 700^{o}C. The thermite reaction $B_2O_3 + Al \leftrightarrow 2B + Al_2O_3$ began when the temperature reached to 875^{o}C. The product boron and unreacted aluminium reacted and yielded as AlB_2 form. At temperature higher than 1000^{o}C, Borates, $Al_4B_2O_9$, $Al_{18}B_4O_{18}$ and AlB_{10} were founded in the alloy microstructure.

The formation and growth behaviour of aluminium boride crystals in an Al-B alloy was analysed by Kayikci et al [20-21]. Al-3 wt. % B master alloy was obtained as a result of reinforcement of B_2O_3 in the alloy. In the experiment, temperature was increased up to 1300°C and waited for 30 minutes then melted alloy was quickly cooled below peritectic temperature which was 1000°C to abstain AlB_{12} particles. In the end of this study high aspect ratio particles were founded in the microstructure of alloy. None-flake shaped particles such as cubic and tetrahedral were come in sight. None-flake particles are not preferred in the manufacturing process of metal matrix composites. Fast cooling was applied to melted mixture to eliminate higher borides.

Fabrication and properties of functionally graded Al/AlB_2 composites was explored by Kayikci and Savas [22]. Centrifugal casting method was used to achieve Al-B cast alloy. 1.2 -1.85 % weight of boron was added to aluminium at 1400°C for 30 minutes. AlB_2 flakes were formed then the cast alloy was re-casted by centrifugal casting method at 800°C. Subsequently, outer side of casting had 11 % AlB_2 rich part, the inner side was lowest 0.02% AlB_2 percentage. Besides, tension tests and hardness tests were executed to samples which have been taken from different part of the casted Al-B alloy. Hardness tests results showed that boron rich part of samples had high hardness values. Tensile strength of the samples had higher values by adding boron. Besides, external region of the cast alloy was brittle fracture but internal region had ductile fracture.

Zhu Nie et al [23] concentrated on production of boron carbide reinforced 2024 aluminum matrix composites by mechanical alloying. B_4C_p / 2024Al composite were produced by mechanical alloying-hot extrusion technology and mechanical properties of the alloy was investigated and compared. In accordance with this study, boron carbide particles were dispersed homogeneously through alloy. The surface of obtained alloy had no defects, any pore and cracks. Mechanical properties of boron carbide reinforced composite had enormous ultimate strength value than unalloyed aluminium.

A study related to processing and mechanical properties of AlB_2 flakes reinforced Al-alloy composites was put into practice by Deppisch et al [24]. AlB_2 crystals strengthened with the volume fracture increased up to 20% in the aluminium copper alloy were successfully fulfilled at 1300°C. AlB_{12} form of higher borides particles were limited thanks to decreasing temperature of 1300°C to room temperature by argon flow quenching process. The particles high aspect ratio of approximately 30 were obtained and equally dispersed in the aluminium alloy. In this examination, 2011 aluminium which have 5-6% wt. copper content and another aluminium alloy which have 5% wt. boron content were chosen. Copper alloy was utilized in order to investigate the effect of copper on the flake formation and make use of high strength performance of copper. Then, 40% wt. Al-B alloy and 60% wt. 2011 Al-Cu alloy were heated in a crucible at 760^{o}C and later melted mixture was casted into moulds which have 5 cm diameter and 7 cm length. Subsequently, heat treatment process was first performed by heating again the casted samples to 1300^{o}C and then it was cooled to 600^{o}C. Finally, the melt temperature was decreased to room temperature. Microstructure observation and mechanical properties of casted AlB_2/Al-Cu alloy were researched and made a comparison with mechanical properties of unreinforced samples.

The paper presented that equally dispersed 10-20 vol. % AlB_2 particles in the AlB_2/Al-Cu alloy was attained from 1360^{o}C by rapid cooling to room temperature and avoided from AlB_{12} formation. AlB_2/Al-Cu alloys displayed high strength and modulus compared with unalloyed samples. The 20 vol. % AlB_2 reinforced alloy showed a 71% increase in strength than the other volume fraction samples. Fracture surfaces of strengthened alloys showed significant AlB_2/Al-Cu interface bonding with no flake de-cohesion and brittle fracture type failures were generally observed during the mechanical tests.

Khatami and Mirdamadi [25] fulfilled a work on the effect of controlled quenching on the aging of 2024 aluminium alloy containing boron. Aluminium and boron powders (B_2O_3) were blended and heated at 800^{o}C. After holding 10 minutes, the melted alloy was casted into steel moulds. Hot rolling process was executed to the cast alloy at a temperature greater than recrystallization temperature to convert it to sheet metal. The specimens were heated for 3 hours at 460^{o}C to eliminate cold working effect. Later, boron reinforced alloys were heat treated at 490^{o}C holding 1 hour and then quenched at 25^{o}C, 50^{o}C, 80^{o}C, 110^{o}C, 150^{o}C, 180^{o}C during different holding times. Subsequently, different kind of heat treatment processes were performed to investigate behaviour of controlled quenching on the mechanical properties.

The work resulted that the optimum aging temperature and time were respectively recorded as 110^oC and 40 minutes. The hardness of the samples increased from 73 Brinell to 136.4 Brinell during 40 minutes by controlled quenching. However, at conventional quenching process, hardness value enhanced from 96 Brinell to 124.3 Brinell in a more time, 90 minutes. Boride particles increased ultimate tensile strength and yield strength than unreinforced.

A study on "Improvement of mechanical properties of AA1070 aluminium by the addition of borax under different holding conditions", Yılmaz and Öztürk produced Al-B alloys by the addition of different amounts of borax powders such as wt. % 5, % 10, % 15, % 20 to 99.70 % pure commercial aluminium. The targeted aim of the study was to investigate the influence of borax addition and holding parameters on the mechanical properties of cast Al-B alloys in an exothermic reaction. This work presents that amount of borax, holding temperature such as 800^oC, 1000^oC and 1200^oC and holding time 30, 45 and 60 minutes, respectively were affecting parameters on the mechanical properties of the Al-B alloys. Mechanical tests such as tensile, impact and hardness tests were performed. According to mechanical test results, tensile strength of the alloy with the adding of borax amount was measured 33% increase than pure aluminium, also 21% increase in impact energy and 54% within hardness measurements. A substantial quantity of aluminium-boride flakes were seen in the microstructural characterization of the samples. Microscopic examinations of alloyed samples show homogeneous distribution of aluminium boride particles in the aluminium matrices [26].

2.3 PLACE OF THIS WORK IN LITERATURE

Literature review about Al-B alloys were searched and analysed. Literature knowledge and experiences from previous studies showed some investigations performed on the different behaviours of Al-B alloys such as type of boron sources and type of production methods but there are not adequate technical achievements how aspect ratio effects the mechanical and physical properties of Al-B alloys. This study attempts to complete the deficiency on effect of the aspect ratio of AlB_2 flakes on mechanical and physical properties of Al-B alloys.

CHAPTER 3

IMPORTANCE OF ALUMINIUM, BORON AND AL-B ALLOYS

3.1 ALUMINIUM

Aluminium is the third most widely found metal in the world. Aluminium has very excellent properties such as low density, high strength, good malleability, easy machining, excellent corrosion resistance and good thermal and electrical conductivity and easy recycles process. Aluminium is very desirable metal in engineering applications, automotive and aerospace industry due to superior mechanical properties.

3.1.1 Physical and Chemical Properties of Aluminium

One of the best known properties of aluminium is that it is light, with a density one third that of steel, 2.700 kg/m3. Aluminium alloys commonly have tensile strengths of between 70 and 700 MPa. The range for alloys used in extrusion is 150 – 300 MPa. Compared with other metals, aluminium has a relatively large coefficient of linear expansion. Aluminium is easily worked using most machining methods such as milling, drilling, cutting, and punching.

Aluminium is an excellent conductor of heat and electricity. An aluminium conductor weighs approximately half as much as a copper conductor having the same conductivity. Aluminium reacts with the oxygen in the air to form an extremely thin layer of oxide. This layer is dense and provides excellent corrosion resistance [27].

Table 3.1 Physical and Chemical Properties of Aluminium [28]

Property	**Value**
Atomic Weight	**:** 26.98 gr/mol
Melting Point	**:** 660.32 ^{o}C
Boiling Point	**:** 2467 ^{o}C
Density	**:** 2.71 g/cm^{3}
Electrical Resistivity	**:** (20 ^{o}C) 28.2 nΩ.m
Thermal Conductivity	**:** 237 W/(m.K)
Heat of Vaporization	**:** 294 kj/mol
Heat Capacity	**:** (25 ^{o}C) 24.2 J/(mol.K)
First Ionization Energy	**:** 577.5 kj/mol
Atomic Radius	**:** 125 pm (0.125 nm)

3.1.2 Aluminium Products and Application Areas

Aluminium alloys are widely used in automobile structures and aerospace vehicles due to their excellent strength, low-weight ratio, good corrosion resistance and easy machinability shown in Figure 3.1.

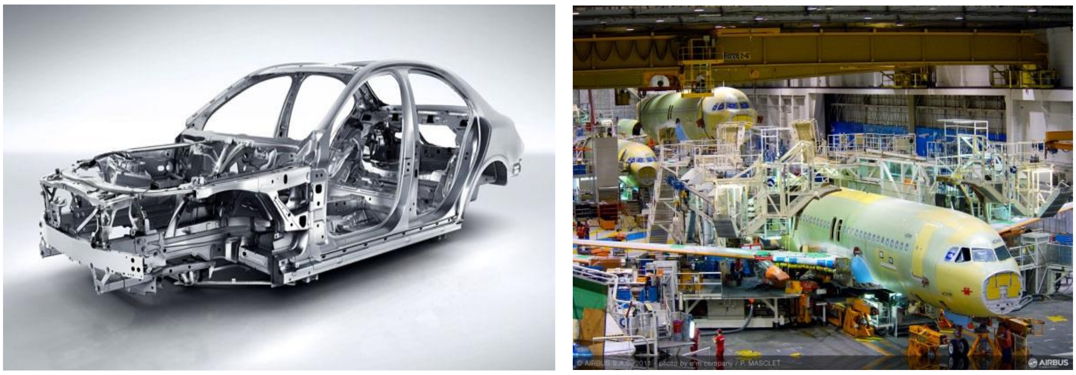


Figure 3.1 Automobile and Aeroplane Body Structures

Structural components manufactured by aluminium, from automotive to aerospace, medical to military industry and transportation to the construction industry, have an important execution area in daily life.

Aluminium alloys are also used in architectural design, marine constructions, truck-trailer body panels and production of electrical wires.

3.2 BORON

Boron is a natural element in the earth crust. In the environment, boron is combined with oxygen and other elements in compounds called borates. Borates are widely found in oceans, sedimentary rocks, coal, shale and some soils. There are over 200 naturally occurring boron containing minerals but the most commercially important and frequently traded minerals (salts, known as borates) are tincal, colemanite, ulexite and kernite [29].

The main commercially important boron minerals:

- Tincal $Na_2B_4O_7.10H_2O$
- Kernite $Na_2B_4O_7.4H_2O$
- Colemanite $Ca_2B_6O_{11}.5H_2O$
- Ulexite $NaCaB_5O_9.8H_2O$
- Datolite $CaBSiO_4(OH)$
- Hydroboracite $CaMgB_6O_{11}.6H_2O$

The main commercial boron compounds are:

- Boric acid H_3BO_3
- Anhydrous boric acid B_2O3
- Anhydrous borax $Na_2B_4O_7$
- Borax pentahydrate $Na_2B_4O_7.5H_2O$
- Borax decahydrate $Na_2B_4O_7.10H_2O$
- Sodium Perborate $NaBO_3.H_2O$

3.2.1 Physical and Chemical Properties of Boron

There are several allotropic forms of boron. Boron compounds can be classified into two groups. These are amorphous boron and crystalline boron. Elemental boron exists as a solid at room temperature, either as black monoclinic crystals as a yellow or brown amorphous powder when impure. The amorphous and crystalline forms of boron have densities of 2.3 and 2.34 g/cm^3, respectively. Physical and chemical properties of boron are given in Table 3.2.

Table 3.2 Physical and Chemical Properties of Boron [30]

Property	Value
Atomic Weight	: 10.811
Boiling Point	: 4002°C
Thermal Expansion Coefficient	: 0.0000083 cm/cm/°C (0°C)
Conductivity	: Electrical: 10^6 Ω.m @25°C Thermal: 0.274 W/cmK
Density	: 2.34 g/cc @ 25°C
Appearance	: Yellow-Brown Nonmetallic Crystal
Elastic Modulus	: Bulk: 320/GPa
Enthalpy of Atomization	: 573.2 kJ/mole @ 25°C
Enthalpy of Fusion	: 22.18 kJ/mole
Enthalpy of Vaporization	: 480 kJ/mole
Hardness	: Vickers=49000 MN m^{-2}
Heat of Vaporization	: 489.7kJ/mol
Melting Point	: 2300°C
Molar Volume	: 4.68 cm^3/mole
Physical State	: Solid (20°C & 1 atm):
Specific Heat	: 1.02 J/gK
Vapour Pressure	: 0.348 Pa@2300°C

3.2.2 Boron Products and Application Areas

Glass industry has highest boron consumption. Boron addition to the glass product in molten form, boron increases the viscosity of the glass and enhances the surface hardness and strength of the final product. Boron oxide is extensively used primarily in borosilicate glass, textile-type and insulating glass fibres.

Boron use in the aeronautics and space industry demonstrates a progressive rise. Advancements in aerodynamics, high speed wing applications and air frames resistant to high temperatures, R&D efforts on low-weight and high-capacity projects have fairly broadened the use of composite materials in the aeronautics and space industry.

Boron regulates glucose diffusion in the cells, growth and division of the cells, and photosynthesis metabolism. Plants can grow and foliate without boron, but losses might occur in fruit or seed production.

In addition to nutrition, boron element is also in our daily life with detergents and cosmetic products. Some types of boron formulas (sodium perborate etc.) are also seen in the detergent industry, home cleaning, personal care products, industrial bleaching, and antibacterial products. Boron is a preferred element in the cosmetics sector as it provides softness, stickiness and endurance to the products [31].

Boron fibres (boron filaments) are high-strength, lightweight materials that are used chiefly for advanced aerospace structures as a component of composite materials, as well as limited production consumer and sporting goods such as golf and fishing rods [32].

General Boron and products application areas:

- Glass and Ceramics Industry
- Aerospace Industry
- Cleaning and Bleaching Industry
- Agriculture
- Metallurgy
- Energy
- Health
- Cement

3.2.3 World Boron Reserves and Importance for Turkey

In the world, Turkey, USA and Russia have the important boron reserves. In terms of total reserve basis on, Turkey has a share of %72.20, the other important country USA is %6.8. Total world boron reserves on the basis of B_2O_3 content are 1.176 million tons. With a share of %72.20, Turkey has total boron reserves of 851 million tons on the basis of B_2O_3 content.

Turkey is the largest producer of boron ore in the world. Important boron minerals of Turkey's are tincal, colemanite and ulexite. Boron minerals contain different amount of B_2O_3 in their structures. The important factor for industrial application of boron minerals are B_2O_3 content [33].

3.3 METAL MATRIX COMPOSITES

In recent days, metal composite materials have been used many different application especially in engineering, automobile, aerospace industry. Metal matrix composites (MMCs) are fabricated by means of unification of two or more different materials, which are form of reinforced particles or continuous-discontinuous fibers by dissolving and dispersion into a molten metal or partially solidified metals as a new material [34].

These advanced and new materials introduce many possibilities for modern material science and advanced manufacturing technologies. The characteristic of MMCs provide more low-economic productions and quick solutions for manufacturing. In engineering application, cost of production, production time and higher mechanical properties are very important factors for solutions in engineering problems. Combination of different materials presents enormous mechanical properties in advanced manufacturing technologies. The composite materials have better and superior specification than each component material.

MMCs have many superior properties such as,

- low density,
- mechanical compatibility,
- chemical compatibility,
- thermal stability,
- high Young’s modulus,
- high compression and tensile strength,
- good machinability,
- higher efficiency and cost.

Metal matrix composites can be classified in various ways. One classification is the consideration of type and contribution of reinforcement components in particle, layer, fibres and penetration composite materials (Figure 3.2). Fibre composite materials can be further classified into continuous fibre composite materials (multi- and monofilament) and short fibres or, rather, whisker composite materials shown in Figure 3.3 [35].

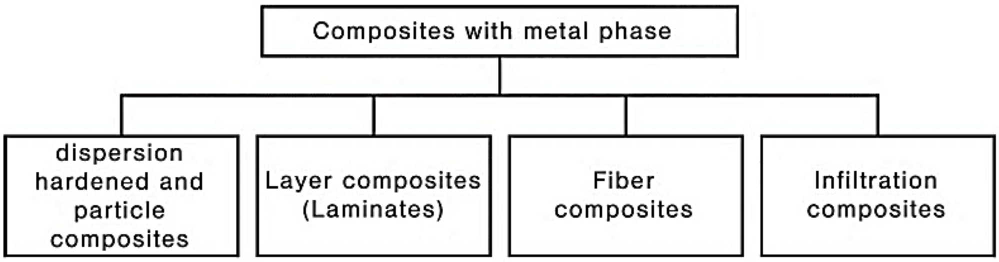


Figure 3.2 Classification of Composite Materials with Metal Matrixes

Main type and contribution of reinforcement components in MMC;

- layer types
- fibre types
- particle types

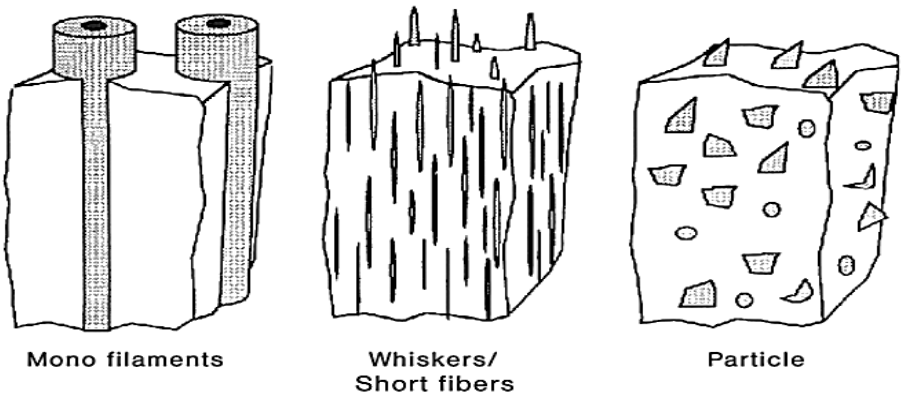


Figure 3.3 Types of Metal Matrix Composites

3.3.1 Fabrication Methods of Metal Matrix Composites

Metal matrix composites are produced by using different kind of production methods. The processes are classified;

- Liquid State Fabrication
- Solid State Fabrication
- Deposition Technique
- In-situ Process

3.3.1.1 Liquid State Fabrication

In liquid state fabrication, materials are melted and dispersed by mechanical stirring into a molten matrix metal, following by solidification. In order to get high level of mechanical properties of the composite, good interfacial bonding (wetting) between the dispersed phase and the liquid matrix must be provided.

The methods of liquid state fabrication of Metal Matrix Composites:

- Stir casting
- Infiltration

3.3.1.2 Solid State Fabrication

In solid state production of MMC, MMC are formed as a result of bonding of matrix metal and dispersed phase due to mutual diffusion occurring between them in solid state at elevated temperature and under high pressure. Powder blending and consolidation is a commonly used method for the preparation of discontinuously reinforced MMCs. In this process, powders of the metallic matrix and reinforcement are first blended and fed into a mold of the desired shape. Blending can be carried out dry or in liquid suspension. Pressure is then applied to further compact the powder (cold pressing).

The methods of solid state fabrication of Metal Matrix Composites:

- Powder Metallurgy
- Diffusion Bonding

3.3.1.3 Deposition Technique

These processes for metal-matrix composite fabrication involve coating individual fibers in a tow with the matrix material needed to form the composite followed by diffusion bonding to form a consolidated composite plate or structural shape. The main disadvantage of using deposition techniques is that they are time consuming. Several deposition techniques are available: immersion plating, electroplating, spray deposition, chemical vapor deposition (CVD), and physical vapor deposition (PVD), spray forming [36].

3.3.1.4 In-situ Process

In these techniques the reinforcement phase is formed in situ. In-Situ Process is a novel technique that does not require any secondary process parameters. In this process the required reinforcing phases are produced by chemical reaction among the appropriate ingredients at suitable temperatures. The composite material is produced in one step from an appropriate starting alloy [37].

3.3.2 Matrix Materials Selection

Wide range of metals can be used for the matrix of the composites whereas lightweight and compatibility with the ceramic reinforcements restricts choice of matrix metal. The common material candidates are Aluminium, Magnesium, Titanium, Copper, Zinc, Nickel and Lithium (Table 3.3).

Table 3.3 The Typical Reinforcements Used in Metal-Matrix Composites [38]

Reinforcement	Matrices
Boron, fiber (including coated)	Aluminium, titanium
Graphite fiber	Aluminium, magnesium, copper
Alumina fiber	Aluminium, magnesium
Silicon carbide fiber	Aluminium, titanium
Alumina-silica fiber	Aluminium
Silicon carbide whisker	Aluminium, magnesium
Silicon carbide particulate	Aluminium, magnesium
Boron carbide particulate	Aluminium, magnesium

Another restriction is that chemical reactions should not take place between matrix and reinforcement. Such a reaction deteriorates reinforcement-matrix interface properties hence the mechanical properties are reduced.

The reinforced metal-matrix composites have enormous importance in aerospace, automotive, electronics and military industry. The reinforced metal-matrix composites had superior properties and reduced cost when compared with pure matrices materials [38].

3.3.3 Aluminium Matrix Composites (AMCs)

Aluminium is the most popular matrix for the metal matrix composites (MMCs). The Al alloys are quite attractive due to their low density, their capability to be strengthened by precipitation, their good corrosion resistance, high thermal and electrical conductivity, and their high damping capacity. Aluminium matrix composites (AMCs) have been widely studied since the 1920s and are now used in sporting goods, electronic packaging, armours and automotive industries. They offer a large variety of mechanical properties depending on the chemical composition of the Al-matrix. They are usually reinforced by Al_2O_3, SiC, C , SiO_2, B, BN, B_4C, AlN may also be considered. The aluminium matrices are in general Al-Si, Al-Cu, 2xxx or 6xxx alloys.

The major advantages of AMCs compared to unreinforced materials are as follows:

- Greater strength
- Improved stiffness
- Reduced density(weight)
- Improved high temperature properties
- Controlled thermal expansion coefficient
- Good thermal and electrical conductivity

- Enhanced and tailored electrical performance
- Improved abrasion and wear resistance
- Control of mass (especially in reciprocating applications)
- Improved damping capabilities.

3.3.4 Fabrication of the Aluminium Matrix Composites

There are many processes viable to fabricate AMCs; they can be classified in: solid - state, liquid-state and deposition processes.

In solid-state processes, the most spread method is powder metallurgy PM; it is usually used for high melting point matrices and avoids segregation effects and brittle reaction product formation prone to occur in liquid state processes. This method permits to obtain discontinuously particle reinforced AMCs with the highest mechanical properties.

In liquid-state processes, one can distinguish the infiltration processes where the reinforcements form a preform which is infiltrated by the alloy melt with pressure applied by a piston or by an inert gas (gas pressure infiltration GPI) and without pressure. In the last case, one can distinguish (a) the reactive infiltration processes using the wetting between reinforcement and melt obtained by reactive atmosphere, elevated temperature, alloy modification or reinforcement coating (reactive infiltration) and (b) the dispersion processes, such as stir-casting, where the reinforcements are particles stirred into the liquid alloy. Process parameters and alloys are to be adjusted to avoid reaction with particles.

In deposition processes, droplets of molten metal are sprayed together with the reinforcing phase and collected on a substrate where the metal solidification is completed. This technique has the main advantage that the matrix microstructure exhibits very fine grain sizes and low segregation, but has several drawbacks: the technique can only be used with discontinuous reinforcements, the costs are high, and the products are limited to the simple shapes that by obtained by extrusion, rolling or forging [39].

3.4 ALUMINIUM BORON ALLOYS

Al–B master alloys are widely used in the production of conductor grade aluminium to remove transition metals reducing the electrical conductivity of aluminium dramatically. Transition metals such as titanium, vanadium, chromium and zirconium are readily precipitated by boron as their borides are more stable than those of aluminium.

These elements are brought into aluminium as impurities with bauxite or from scraps. The transition elements reduce the electrical conductivity of aluminium dramatically. To overcome this problem, boron is used to precipitate these impurities by forming borides of transition metals in aluminium which do not contribute to a major reduction in the electrical conductivity. Al–B master alloys are also used in the in-situ fabrication of aluminium matrix composites. Al–B master alloys can be used for the in situ fabrication of boride-reinforced aluminium matrix composites for the same reason. However, the most promising application of Al–B master alloys is in grain refining which has become a standard melt treatment practice in aluminium foundries world-wide with well documented technical and economic benefits [40].

3.5 AL-B PHASE DIAGRAM AND PERITECTIC TEMPERATURE

The analysis of Al-B binary system is important for boron treatment process. Boron has limited solubility in solid aluminium (Figure 3.4). As seen from the Al-B binary phase diagram, solubility of boron in liquid aluminium is very small at low temperatures. At 1200 °C solubility ratio is about 2% by weight. This ratio is below necessary critical reinforcement ratio for fabrication of AlB_2/Al. Many studied were executed on phase diagrams for the Al-B alloys.

Boron reacts with aluminium to form Al-B compounds such as AlB_2 and AlB_{12}. All of them verified the existence of phases, AlB_2 and AlB_{12}. Also they showed the execution of a peritectic transformation during cooling of liquid aluminum to solid form. In this reaction, AlB_{12} particles transform to AlB_2 [41].

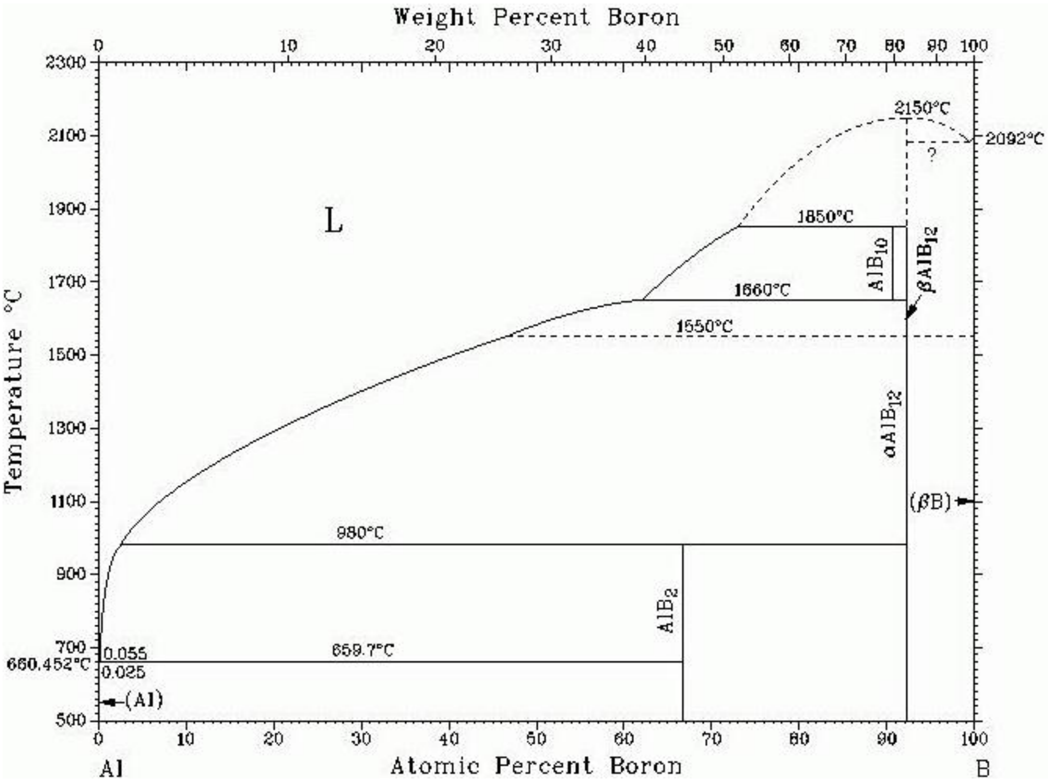


Figure 3.4 Aluminium Boron Phase Diagram [42]

In order to increase reinforcement ratio, Deppisch [24] increased reinforcement ratio up to %20 by holding and filtering Al-B alloy %4 by volume at 700 ^{o}C in furnace. Economy [13] succeeded increment of reinforcement ratio up to %30 by using the same method. Duque et all [45] accomplished rising reinforcement ratio up to %20 by using centrifugal casting method.

Boron is reduced by melting aluminium during an in–situ exothermic reaction which is $Al_{(L)}+AlB_{12} \leftrightarrow AlB_2$ at peritectic temperature 975^{o}C in aluminium boron phase diagram. Borax powders are dissolved and dispersed in molten aluminium, yielding aluminium borides which are, AlB_2 in hexagonal and AlB_{12} in tetragonal crystal structures, respectively.

According to Al-B phase diagram, AlB_{12} is shown at the temperatures above peritectic temperature of 975^{o}C. AlB_2 crystals are shaped without higher borides such as AlB_{12} which is thought to confine strength of the Al-B alloys when reaction temperature passes over peritectic temperature and then melting is cooled to room temperature. AlB_{12} crystals transform to AlB_2 flakes [43].

CHAPTER 4

EXPERIMENTAL PROCEDURE AND SETUP

4.1 INTRODUCTION

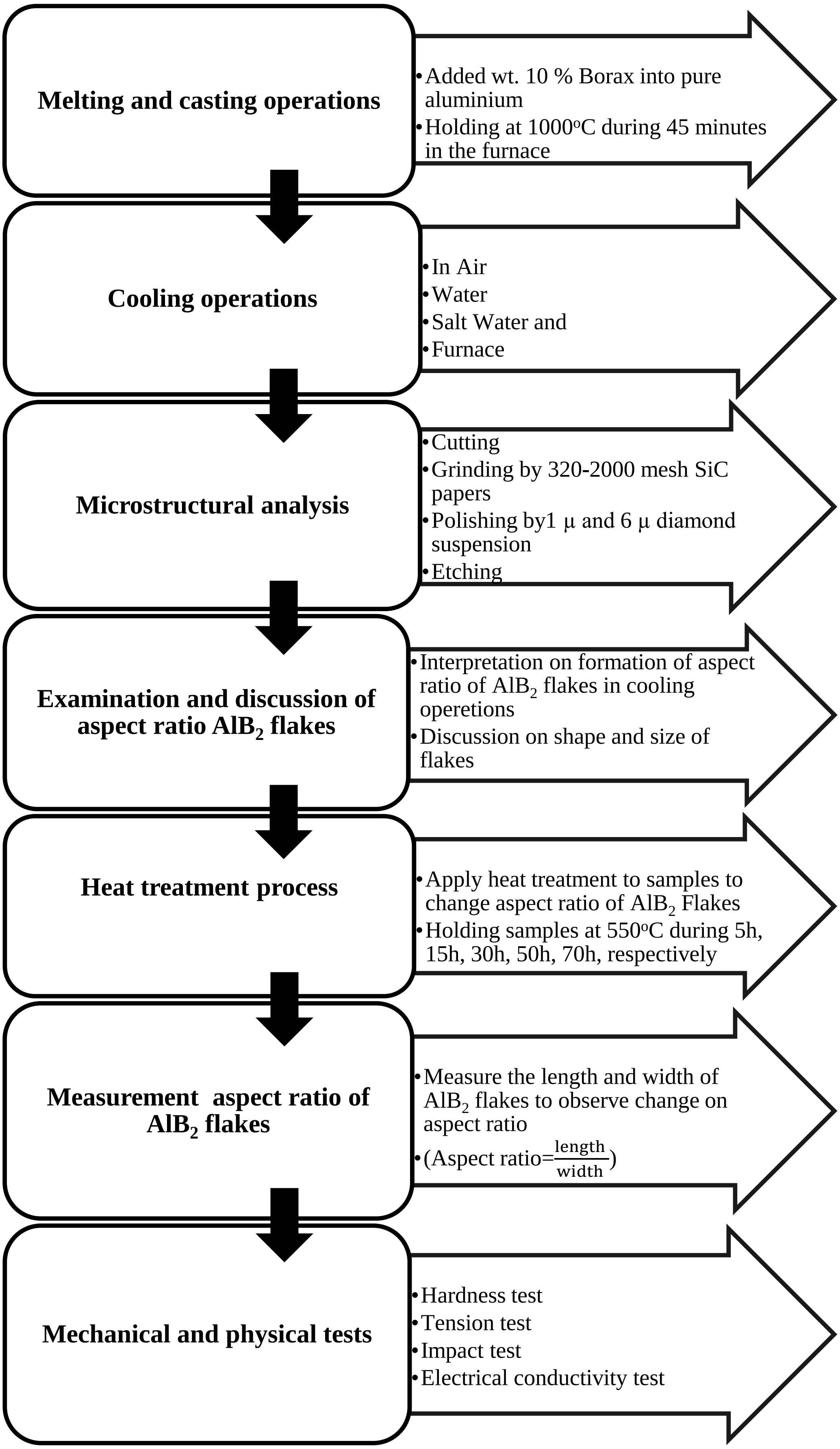
In this study, pure aluminium and 10% wt. borax powders ($Na_2B_4O_7.10H_2O$) were blended in a silicon carbide crucible. Aluminium and borax mixture was heated up to 1000°C which is above peritectic temperature in Al-B phase diagram with 45 minutes to obtain AlB_2 compounds.

Boron was dissolved by melting aluminium during an in–situ exothermic reaction which is $Al_{(L)}+AlB_{12} \leftrightarrow AlB_2$. And then metallographic processes were performed to cast Al-B alloys for microstructure examination.

At the end of these stages, cast Al-B samples were exposed to mechanical tests such as Brinell hardness test, tensile test and impact energy test and physical tests such as electrical conductivity of the Al-B alloys.

In this chapter, general experimental procedure of fabrication aluminium boron alloys is mainly explained in Table 4.1. Microstructural examination processes and performed mechanical and physical tests were explained in details.

Table 4.1 Flow Chart of the Study



4.2 MATERIALS

In the experimental method, 99,70% AA1070 pure aluminum, borax powder ($Na_2B_4O_7.10H_2O$) (Figure 4.1) and also synthetic cryolite (Na_3AlF_6) as a reaction activator, gas removal tablet (Aldegas 100) bought from Seydisehir Aluminum Inc. (Turkey) and boric acid (H_3BO_3) as a reaction preventive block between melt and crucible, which obtained from Eti Maden Works (Turkey) were utilized to fabricate Al-10 wt.% B alloys. Chemical composition of AA1070 pure aluminum is displayed in Table 4.2.

Table 4.2 Chemical Composition of AA 1070 Aluminium (%) [44]

Aluminium, Al	99.7
Copper, Cu	0.04
Iron, Fe	0.25
Magnesium, Mg	0.03
Manganese, Mn	0.03
Silicon, Si	0.2
Tinanium, Ti	0.03
Vanadium, V	0.05
Zinc, Zn	0.04
Other	0.03

Figure 4.1 Aluminium and Borax Powders

4.3 FABRICATION METHOD OF THE AL-B ALLOYS

Pure aluminium and 10% wt. borax powders were blended in the silicon carbide crucible which was covered by boric acid (H_3BO_3) to prevent reaction and adhere of the slag to the crucible. Synthetic cryolite (Na_3AlF_6) was added as a reaction activator and grain refiner. Gas removal tablet (Aldegas 100) was also added to melting to prevent gas porosity.

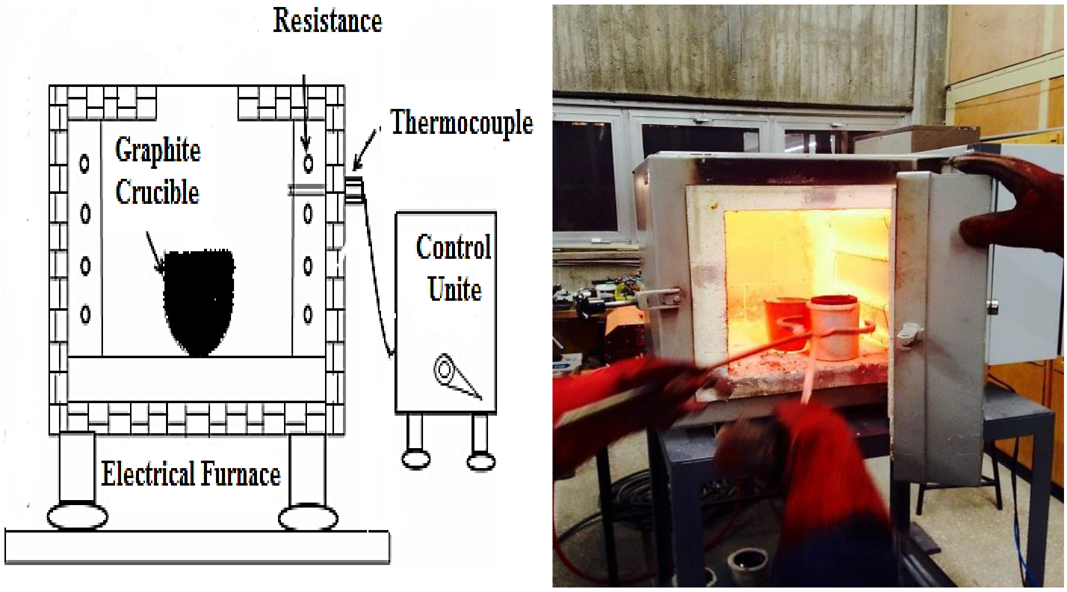


Figure 4.2 Experimental Set up of Casting Operation and Electrical Furnace

Then furnace was heated up to 1000^{o}C and stirred by a carbide rod to provide homogenous disperse of borax in the alloy. When the temperature reached to 1000^{o}C, melted mixture was waited at the electrical resistance furnace during 45 minutes. Melted aluminium and borax mixture was removed from furnace (Figure 4.2). Before casting alloy to the steel molds (Figure 4.3), the slug which is protective layer from atmospheric effects on the surface of melted aluminium, was cleared away and casted.

Figure 4.3 Silicon Carbide Crucible and Permenant Steel Molds

4.4 HEAT TREATMENT PROCESS

The cast Al-B alloy specimens were first cooled in air. The cast alloys were cut into pieces. Then, samples were placed into the furnace (Figure 4.4). Heat treatment process was carried out by heating in the furnace at 520 °C during different holding times 5h, 10h, 30h, 50h and 70h to obtain different aspect ratio of AlB_2 particles. At the end of the each holding time, samples were removed from the furnace and microstructural examinations were executed.

Figure 4.4 Samples Prepared for Heat Treatment Processes

4.5 MICROSTRUCTURE EXAMINATION

The cast Al-B alloys were divided into pieces to observe microstructures. The samples were ground by 320, 600, 1000 and 2000 mesh SiC papers and polished by 6 μ and 1 μ diamond suspension in the Metkon Forcipol 2V Grinding and Polishing Machine shown in Figure 4.5.

Figure 4.5 Grinding and Polishing Machine

Later, colloidal silica solution was applied to polished surfaces to obtain high-quality pictures from surfaces. Subsequently, etching was performed by using 0.5 hydrofluoric acid (HF) during 45 seconds to see surface structures in details. The microstructure pictures were displayed by Nikon MA-100 Optical Microscope shown in Figure 4.6.

Figure 4.6 Optical Microscope

Scanning electron microscope was used to display the dissolved AlB_2 flakes in aluminium matrices. Rectangular, cylindrical and hexagonal shaped AlB_2 flakes were clearly observed using Scanning Electron Microscope JEOL JSM-639OLV shown in Figure 4.7.

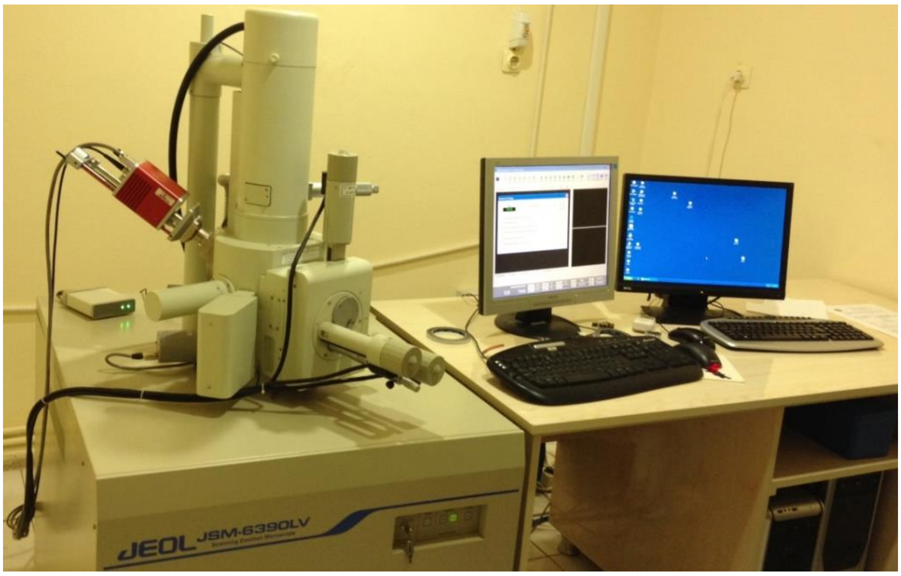


Figure 4.7 SEM and Control Unite

4.6 MECHANICAL TESTS

4.6.1 Hardness Test

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. The usual method to achieve a hardness value is to measure the depth or area of an indentation left by an indenter of a specific shape, with a specific force applied for a specific time. There are large varieties of methods used for determining the hardness of materials, but three popular standard test methods are applied for expressing the relationship between hardness and the size of the impression, these are Brinell, Vickers, and Rockwell hardness test.

Brinell Hardness Test were performed to heat treated samples to measure hardness values by applying 62,5 kg load with a 2,5 mm diameter ball in the Digirock Hardness Tester in Figure 4.8. Hardness measurements were recorded and then change of hardness values were calculated and compared with heat treated samples and pure aluminium casted at 1000^{o}C for 45 minutes.

Figure 4.8 Hardness Test Machine

4.6.2 Tensile Test

Tensile test is the most common used mechanical test to measure the mechanical properties of the materials. The major parameters that describe the stress-strain curve obtained during the tension test are the tensile strength (UTS), yield strength or yield point (σ_y), elastic modulus (E), percent elongation ($\Delta L\%$) and the reduction in area (RA%). Toughness, Resilience, Poisson's ratio (ν) can also be found by the use of this testing technique.

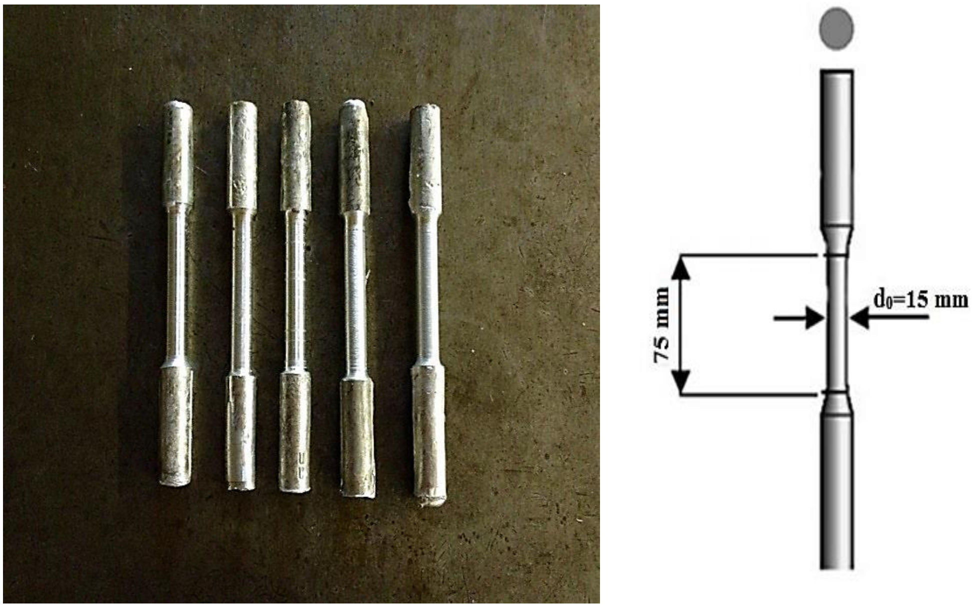


Figure 4.9 Tensile Test Specimens

The central portion (gage portion) of the length is usually of smaller cross section than the end portions. This ensures that failure will occur at a section where the stresses are not affected by the gripping device.

In this study, tensile test was performed to determine ultimate tensile strength (UTS) of the specimens. Cylindrical specimens were prepared according to TS 138 standard to perform the test.

Samples were prepared by turning machine with a diameter of 15 mm and gauge length 75 mm (Figure 4.9). Tensile test of the samples were carried out by using Dartec universal testing machine having a capacity of 60 tonnes in Figure 4.10.

Figure 4.10 Tensile Test Machine

4.6.3 Impact Test

Impact tests are performed to measure the resistance to failure of a material to a suddenly applied force. This test measures the impact energy or the energy absorbed prior to fracture. In this study, Charpy test method was applied to specimens which have a length of 55 mm, a width of 10 mm and a height of 10 mm with a V-shaped notch, 2mm deep, with 45° angle and 0.25mm radius along centre of the specimens (Figure 4.11). Charpy impact test is made up of a hammer which is held at a defined height and a fixed v-notched test specimen applied sudden force.

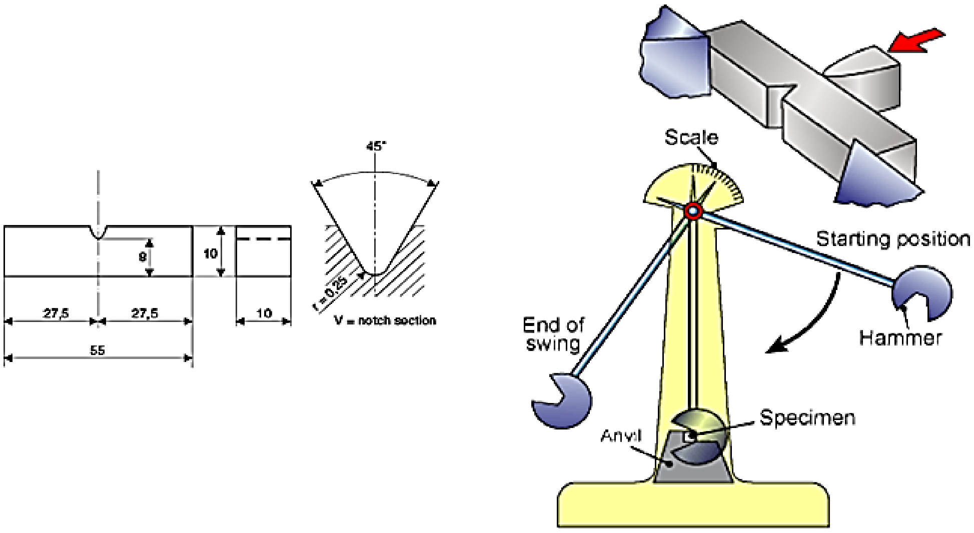


Figure 4.11 Impact Test Set up and Specimen

Impact energy was measured according to height that hammer is released. The work piece was exposed to plastic deformation during impact energy test. The total impact energy depends on the size of the test specimen.

Figure 4.12 Impact Energy Test Machine

In this study, three specimens were prepared for each condition. Tinius Tolsen test machine in Figure 4.12 was used to measure impact energy of the Al-B alloys. Specimens were prepared for each condition by using milling machine.

4.7 PHYSICAL TEST

4.7.1 Four-Point Probe Conductivity Test

The electrical resistivity of aluminium samples were measured by using a four-point probe method. Four conductive wires and samples were placed on a non-conductive block shown in Figure 4.13.

Wires attached to aluminium samples which have 10 mm x 10 mm dimensions and 1.5 mm thickness. The two outer leads were connected to a precision current source and the two inner leads are used to measure voltage drop.

Conductivity was calculated from the measured resistance and dimensions of the specimen. Ohm’s law (V = IR) allows the resistance (R) to be determined. Coupled with cross-sectional area (A) measurement of the sample and the known gage length (L), the resistivity is = R.A/L and conductivity is= 1/resistivity.

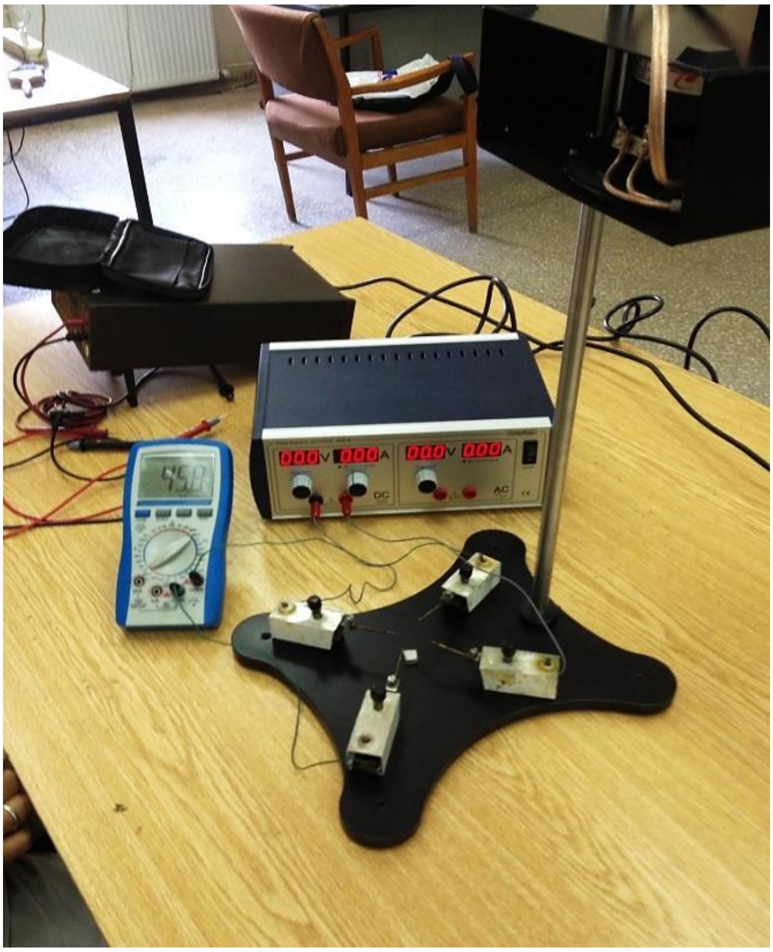


Figure 4.13 Four-Point Probe Conductivity Test Set-up

CHAPTER 5

RESULTS AND DISCUSSION

5.1 INTRODUCTION

In this study, pure aluminium and borax powders were blended and casting operations were performed. Comprehensive microstructural observation was executed to observe distribution of borax particle, size and shape of AlB_2 flakes in aluminium matrices.

The effect of aspect ratio of AlB_2 flakes on mechanical and physical properties of aluminium boron alloys were investigated. In order to get different aspect ratio of AlB_2 particles different processes were done. The cast alloys were cooled in four cooling conditions such as water, salt-water solution, air and furnace.

The effects of cooling conditions were observed on aspect ratio of the particles. Finally heat treatment process was tried to change the aspect ratio of particles.

In this chapter, detailed microstructural studies were presented by using optical microscope. The performed processes to change the aspect ratio of particles were shown and discussed. Mechanical test such as tensile, hardness and impact test, physical tests results and graphs were presented and discussed.

5.2 COOLING CONDITIONS AND PROCESSES

5.2.1 Effect of Cooling Conditions and Rates on Aspect Ratio of Flakes

A series of experimental trials of wt. %10 borax reinforced in Al-B alloy at 1000^oC were carried out to compare the effects of cooling conditions on the microstructural properties of the alloys.

The effect of cooling conditions on the aspect ratio of AlB_2 flakes were observed by microscopically examination. Sample 1, 2, 3 were cooled directly and fast with a 2.5^oC / min cooling rate in air, water and wt. %10 salt water, respectively. Sample 4 was again replaced in the furnace after casting process and this sample was heat-treated at 600^oC in the furnace during 30 minutes after casting shown in Figure 5.1. Then furnace was turned off, sample 4 was held in the furnace to being cooled slowly with a 1^oC/min cooling rate in the furnace. The targeted aim was to search effect of fast and slow cooling on shape, size and aspect ratio of AlB_2 flakes.

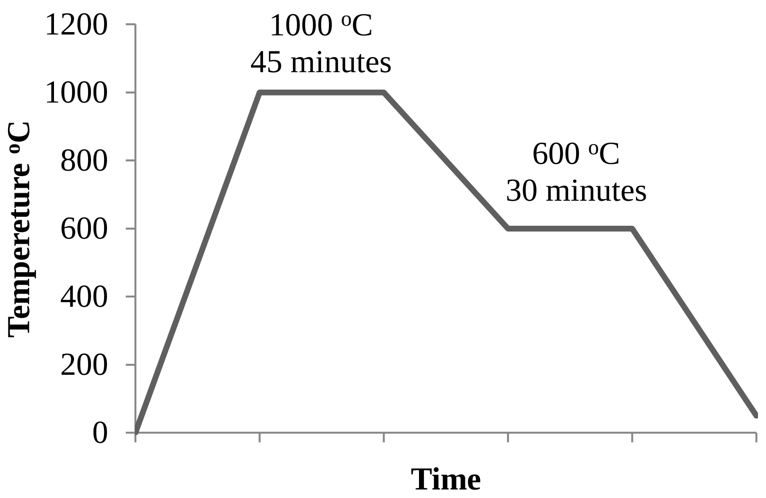


Figure 5.1 Cooling in Furnace

Cooling conditions affect the shape and size of the AlB_2 particles and flakes. In the microstructure of Al-B alloys changed during cooling operations in different cooling conditions such as air, water, salt water solution and slow cooling process in the furnace. After these parameters were applied to sample specimens, particle and flakes size had different aspect ratio.

All these parameters and conditions were demonstrated according to cooling types, method and heat treatment process shown in Table 5.1.

Table 5.1 Experimental Procedure of Group A

GROUP A						
Sample No	**Holding Temp. of Casting**	**Holding Time of Casting**	**Borax Amount in Al-B Alloy**	**Cooling Method**	**Holding Temp. of Heat Treatment**	**Holding Time of Heat Treatment**
1	1000ºC	45 minutes	wt.%10	Air	-	-
2				Water	-	-
3				Salt Water	-	-
4				Furnace	600ºC	30 Minutes

Sample 1 was cooled in the air and it has generally high aspect ratio AlB_2 flakes. When microstructures of the Sample 1 were examined, lengths of flakes are greater and widths of flakes are thinner in Figure 5.2.a. There are many cylindrical flakes in the microstructure. Also rectangular shape AlB_2 flakes are seen in aluminium matrices.

Sample 2 was cooled in the water and it has high aspect ratio AlB_2 flakes in the microstructure examination in Figure 5.2.b. Apart from the flake shape; rectangular, hexagonal and cubical shaped particles were observed in the microstructure. These hexagonal shape AlB_2 flakes have greater mechanical properties due to orientation of crystal structure and amount of the incorporation of AlB_2 flakes. Appearance of these particles is connected to the formation of higher boride particles above the peritectic transformation temperature of 975℃ [12].

The Sample 3 was cooled in the wt. %10 salt in water. The shapes of flakes are cylindrical, rectangular and also hexagonal in Figure 5.2.c. The aspect ratio flakes of Sample 3 are lower than cooled in air. Flakes are generally thicker and shorter.

Sample 4 was prepared in a different manner. The cast alloy was placed into the furnace after casting then it was held at 600^{o}C during 30 minutes. The objectives of heat treatment are to monitor the effect on flake size, shape and aspect ratio of AlB_2 particles. Microstructure of Sample 4 shows higher borides of AlB_{12}. It is observed that particles were accumulated together and very close to each other. They have low aspect ratio than the samples cooled in air, water and salt water solution. Holding in the furnace, the heat treatment process shortened the flakes length.

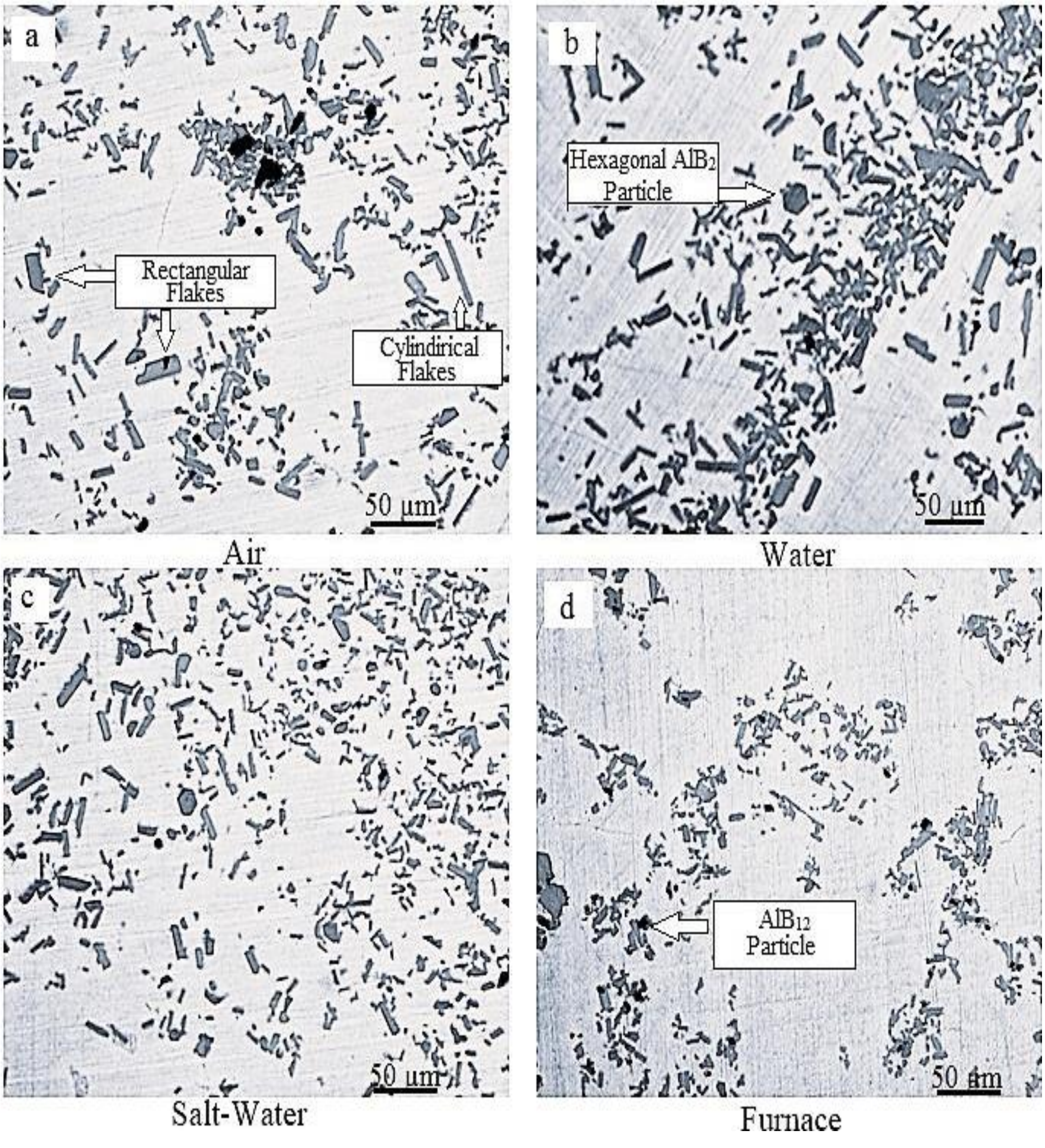


Figure 5.2 The Effect of Coolants on the Microstructure of Al-B Alloy;
a)Cooling in Air b) Cooling in Water c) Cooling in wt. 10% Salt-Water Solution
d) Cooling in Furnace

As a result of the first experimental trials, it is realized that cooling conditions are playing an important role on AlB_2 flakes characterization. As seen in Figure 5.2, fast cooling leads formation of high aspect ratio flakes. The samples cooled in water and air, have higher aspect ratio flakes. And also hexagonal shaped AlB_2 flakes are seen widely in microstructure of sample cooled by water. Fast cooling rate and cooling in water are coming forward for manufacturing high aspect ratio AlB_2 flakes.

The presence of higher borides AlB_{12} particles before formation of AlB_2 should be avoided by raising the cooling rate of the casted alloys. AlB_{12} particles are converted to AlB_2 by peritectic reaction of $AlB_{12} + Al_{(liq.)} \leftrightarrow AlB_2 + Al_{(liq.)}$ from 1000 °C to room temperature. AlB_{12} particles are reported to be brittle and they decrease the properties of the in-situ Al-B reinforced alloys [43].

5.3 HEAT TREATMENT PROCESSES

5.3.1 Determination of Optimum Heat Treatment Process on Aspect Ratio of AlB_2 Flakes

In the Trial Group B, % 99.7 purity aluminium and wt. % 10 borax powders were blended and casted at 1000^{o}C during holding 45 minutes (Figure 5.3) and alloys were cooled in air, water and furnace. In the former trials, salt water solution was tried but effect of this solution didn't change the aspect ratio effectively it was similar to water's effect. So this parameter was not used as a cooling parameter for these trials. Water, air and furnace were preferred as coolants.

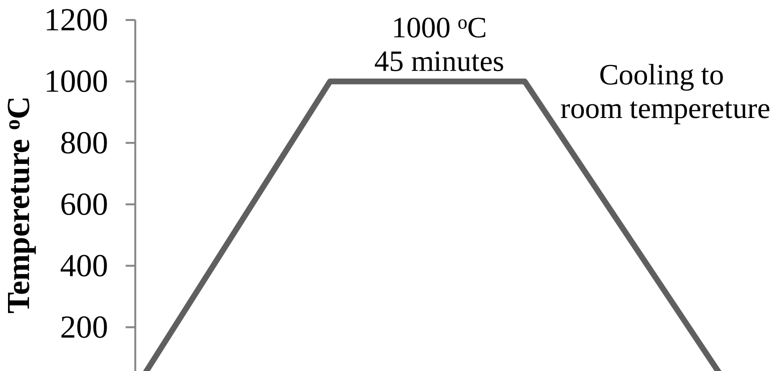


Figure 5.3 Experimental Procedure of Cooling Method

Three group aluminium boron alloys which have a length of 12 cm and diameter of 3 cm were produced with an endothermic in-situ reaction. Samples were cut into pieces which have height of 3 cm and 12 different samples were obtained in Figure 5.4. All parameters and experimental processes performed to sample are presented in Table 5.2.

Table 5.2 Experimental Procedure of Group B

GROUP B						
Sample No	**Holding Temp. of Casting**	**Holding Time of Casting**	**Borax Amount in Al-B Alloy**	**Cooling Method**	**Holding Temp. of Heat Treatment**	**Holding Time of Heat Treatment**
1	1000ºC	45 minutes	wt. %10	Air	550 ºC	1 hour
2				Air	550 ºC	2 hours
3				Air	550 ºC	3 hours
4				Air	550 ºC	4 hours
5				Water	550 ºC	1 hour
6				Water	550 ºC	2 hours
7				Water	550 ºC	3 hours
8				Water	550 ºC	4 hours
9				Furnace	550 ºC	1 hour
10				Furnace	550 ºC	2 hours
11				Furnace	550 ºC	3 hours
12				Furnace	550 ºC	4 hours

After casting operation, heat treatment operation on the cast Al-B alloy was conducted by putting the samples in the furnace. This heat treatment consists of the following steps:

- Replacing the samples in the furnace again and heating them to a temperature of 550℃.

- Holding the cast Al-B alloys at temperature of 550℃ for periods of 1h, 2h, 3h and 4h, respectively.

- Samples finally cooled in air, water and furnace.

Figure 5.4 Samples for Heat Treatment Process

After holding at 550ºC in the furnace, samples were cooled in the air. In order to display the microstructural characterisation of samples; grinding, polishing and etching operations were executed. As seen microscopic examination, non-heat treated samples have thinner AlB_2 flakes having long length. The flakes have high aspect ratio seen in Figure 5.5.a. However, widths of the AlB_2 flakes were very weak so it is thought that they are very strengthless due to being thinner flakes.

When holding time was increased in all cooling conditions, widths of the AlB_2 flakes were increased. However, whenever holding time reached to 4 hours, aspect ratio of the flakes transformed to low aspect ratio. The cylindrical and rectangular shape of flakes converted to round and circular form during 4 hours holding time as seen in the Figure 5.5.b.

According to holding time in the furnace and cooling rate, it was shown that heat treatment processes converted the high aspect ratio (length/ width) AlB_2 flakes to low aspect ratio flakes as seen in the microstructure pictures in Figure 5.5.

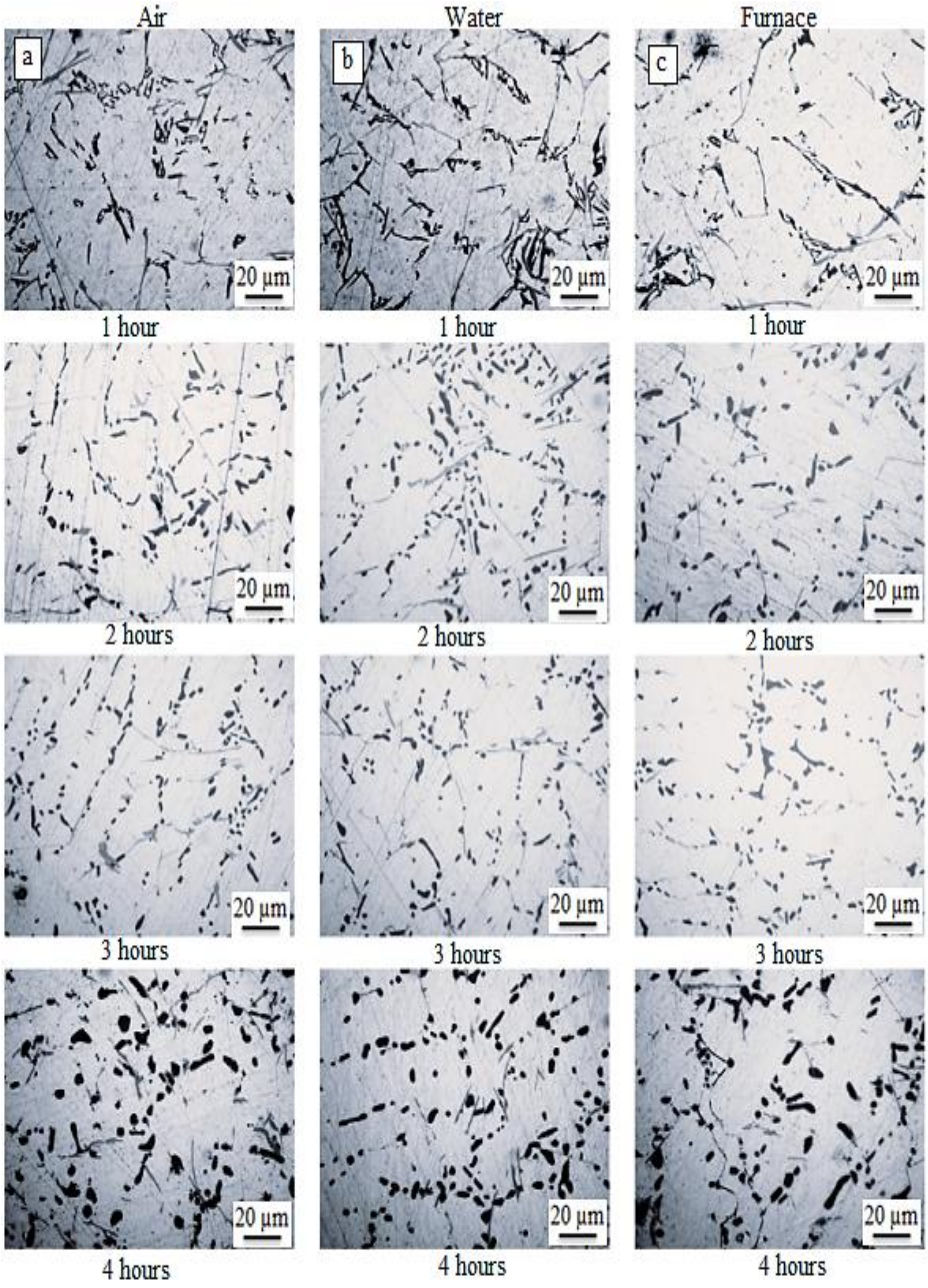


Figure 5.5 Change of Aspect Ratio AlB_2 Flakes with respect to Holding Time

5.3.2 Effect of Increased Holding Time in the Al $_{(solid)}$ + AlB_2 region on Aspect Ratio of AlB_2 Flakes

Another trial set was conducted in order to produce Al-B alloys which have different aspect ratio AlB_2 flakes, it was decided that heat treatment time should be increased by rising the holding time in the furnace to change the aspect ratio of the flakes. In the last stage of this study, water was preferred as a coolant due to effect of formation on high aspect ratio flakes. Because, fast cooling from 1000°C to room temperature is required production of the high aspect ratio AlB_2 flakes.

According to Al-B phase diagram, temperature of 550°C which is lower than melting point of aluminium 660°C in the Al $_{(solid)}$ + AlB_2 region was chosen for aging process shown in Figure 5.6.

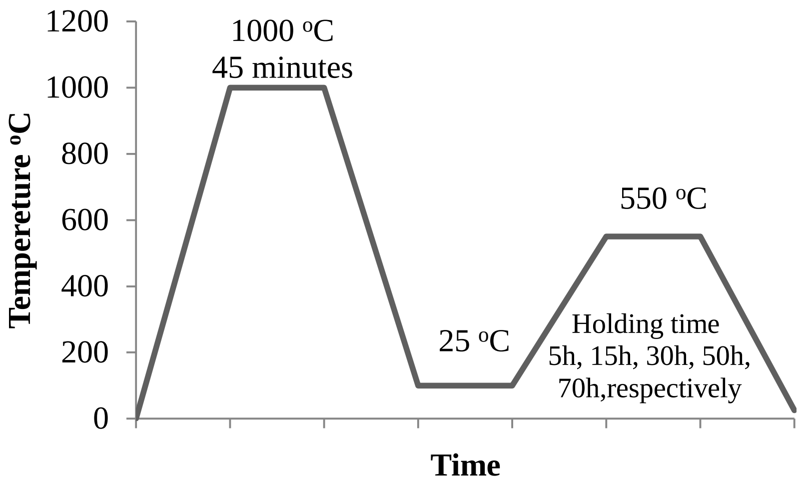


Figure 5.6 Experimental Procedure and Heat Treatment Process

After casting process, samples were cooled in water and they were heat-treated in the furnace at 550°C during different holding times 5h, 15h, 30h, 50h and 70h, respectively. Experimental processes were presented in Table 5.3.

Microstructure analysis in Figure 5.7 shows that widths of the AlB_2 flakes depend on increasing holding time in the furnace at 550°C. Non-heat treated sample in Figure 5.7 (a) AlB_2 flakes were rod-shaped structures which have long length and very thin width. Figure 5.7 (b) and (c), when holding time reached to 5 or 15 hours, these thinner flakes transformed to thicker AlB_2 flakes and they still continued to save long length in these holding times. General particle shapes were rectangular, cylindrical and hexagonal. Moreover, thinner flakes have high aspect ratio (length/width) during 15 hours holding time.

Table 5.3 Experimental Procedure of Group C

GROUP C						
Sample No	**Holding Temp. of Casting**	**Holding Time of Casting**	**Borax Amount in Al-B Alloy**	**Cooling Method**	**Holding Temp. of Heat Treatment**	**Holding Time of Heat Treatment**
C0	1000ºC	45 minutes	wt. %10	Water	550 ºC	0 hour
C5				Water	550 ºC	5 hours
C15				Water	550 ºC	15 hours
C30				Water	550 ºC	30 hours
C50				Water	550 ºC	50 hour
C70				Water	550 ºC	70 hours

However, holding time exceeded 15 hours, cross sectional width of the flakes became larger and length of flakes transformed to shorter height in Figure 5.7 (d), (e) and (f) respectively. Circular shaped particles increased with increasing holding time to 50 hours. Aspect ratio flakes started to decrease. Particles were generally round and circular shaped.

Length and width of the flakes were almost equal size. This situation caused the decrease of the aspect ratio and approaching to 1. In production of AlB_2 flakes, high aspect ratio flakes are transformed to low aspect ratio particles based on decreasing the length and an increment in the width of the AlB_2 flakes.

As a result of experimental trials and casting operations; it is concluded that cooling condition and heat treatment duration were critical parameters for formation AlB_2 flakes and transformation of low aspect ratio flakes to high aspect ratio AlB_2 flakes in Al-B alloys. Cooling conditions and heat treatment clearly effect the length and width of the flakes. During cooling conditions and heat treatment processes, higher aspect ratio AlB_2 flakes transformed to lower aspect ratios.

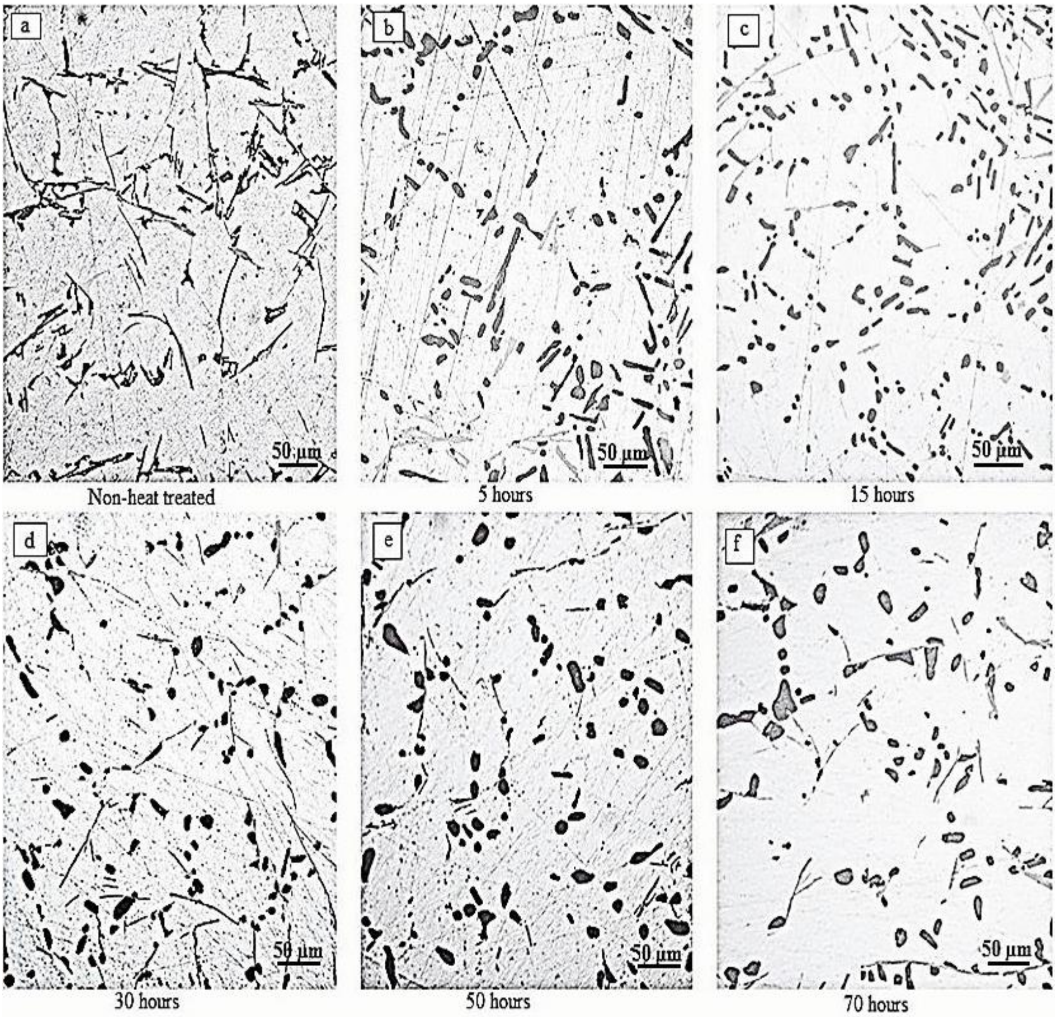


Figure 5.7 Change of Aspect Ratio AlB_2 Flakes with respect to Heat Treatment Time

5.4 AVERAGE ASPECT RATIO MEASUREMENT OF ALB_2 FLAKES

In order to observe the change on the aspect ratio of flakes, average aspect ratios of samples were measured by using “Cameram Software” in Nikon MA-100 Optical Microscope. The average measurements were presented in Table 5.4.

Table 5.4 Average Aspect Ratio Measurements

	C0			C5		
	L(μm)	**W(μm)**	**L/W**	**L(μm)**	**W(μm)**	**L/W**
Meas.1	789	12	65.75	480	59	8.14
Meas.2	986	11	89.64	591	70	8.44
Meas.3	875	13	67.31	431	95	4.54
Meas.4	986	13	75.85	217	56	3.88
Meas.5	1134	12	94.5	160	42	3.81
Meas.6	1098	11	99.82	230	60	3.83
Meas.7	897	10	89.7	470	71	6.62
Meas.8	873	12	72.75	350	58	6.03
Meas.9	876	13	67.38	444	83	5.35
Meas.10	1357	17	79.82	532	79	6.73
			80.25			**5.74**

	C15			C30		
	L(μm)	**W(μm)**	**L/W**	**L(μm)**	**W(μm)**	**L/W**
Meas.1	639	51	12.53	484	67	7.22
Meas.2	535	56	9.554	609	110	5.54
Meas.3	457	70	6.529	738	93	7.94
Meas.4	352	62	5.677	336	108	3.11
Meas.5	371	85	4.365	381	108	3.53
Meas.6	473	57	8.298	401	81	4.95
Meas.7	333	61	5.459	493	209	2.36
Meas.8	414	79	5.241	376	80	4.7
Meas.9	510	38	13.42	284	112	2.54
Meas.10	327	60	5.45	364	95	3.83
			7.65			**4.57**

	C50			C70		
	L(μm)	**W(μm)**	**L/W**	**L(μm)**	**W(μm)**	**L/W**
Meas.1	632	171	3.696	681	130	5.24
Meas.2	579	108	5.361	414	87	4.76
Meas.3	552	157	3.516	549	222	2.47
Meas.4	392	91	4.308	244	70	3.49
Meas.5	367	161	2.28	399	126	3.17
Meas.6	240	133	1.805	404	153	2.64
Meas.7	271	173	1.566	401	185	2.17
Meas.8	357	97	3.68	237	143	1.66
Meas.9	262	77	3.403	271	153	1.77
Meas.10	402	92	4.37	306	216	1.42
			3.40			**2.88**

During measurement of average aspect ratio of flakes in the samples, 5 average measurements were obtained by analysing 10 equal-sized different area of each sample.

As a result of average aspect ratio measurements, non-heat treated sample C0 has the higher aspect ratio of 80.3 but flakes in this sample were very thin and weak. Apart from sample C0, sample C15 has the highest aspect ratio of 7.65 and sample C70 has the minimum aspect ratio of 2.88. When aspect ratios were compared; if holding time was increased in the furnace, aspect ratio of flakes decreased and particles got closer to each other's. That is, longer flakes having high aspect ratio particles transformed to circular particles having low aspect ratio particles.

5.5 MECHANICAL PROPERTIES

Hardness, tensile and impact tests were executed to investigate effect of the aspect ratio of AlB_2 flakes on the mechanical properties of the Al-B alloys. Test result were presented and discussed.

5.5.1 Hardness Test Results

As a result of hardness test, hardness values of Al-B alloys were presented in Table 5.5. Maximum hardness value of 65.1 HB was measured from sample C15 and minimum hardness measurement of 56.8 HB was recorded on the surface of non-heat treated sample C0 in the Al-B alloy.

It is seen that, when holding time of heat treatment is increased, hardness values of alloys increases during 15 hours. After higher holding time of 15 hours, the hardness values of samples are decreased.

When heat treatment duration reaches to 30 hours in the furnace, hardness of C30 decreased to 60.83 HB. During higher holding times, hardness values of C50, C70 have a decrease and values were 59.34 HB and 58.28 HB, respectively.

In order to compare relationship between hardness and aspect ratio of AlB_2 flakes, hardness values with respect to aspect ratio is demonstrated in Figure 5.8. Non-heat treated sample C0 has the maximum aspect ratio of 80.25 HB but this sample has the lowest hardness value of 56.83 HB.

It was thought that cause of this result is related to being AlB_2 flakes have very thin and too long structure. These flakes are much fragile and weak than thicker ones.

Table 5.5 Hardness Test Results (HB)

No	C0	C5	C15	C30	C50	C70
Meas.1	62.4	64.7	65.8	64.9	65.6	62.8
Meas.2	60.8	64.1	65.4	65.8	61.4	58.8
Meas.3	58.6	65.1	68.2	56.2	68	63.1
Meas.4	63.3	65.8	68	65.3	64.3	62.4
Meas.5	64.3	65.4	68.6	65.3	54.1	62.4
Meas.6	58.4	57.1	60.6	55.7	45.7	53.2
Meas.7	44.6	69.4	67.6	59.6	56.7	41.8
Meas.8	58.8	58.4	57.5	59	57.3	60.4
Meas.9	45.1	63.3	64.9	57.9	62.8	62
Meas.10	52	64.5	64.3	57.9	57.5	55.9
Average	56.8	63.78	65.1	60.83	59.34	58.28

Relationship between aspect ratio and hardness has an almost linear change according to data on the graph shown in Figure 5.8.

It was observed from hardness test results that aspect ratio of the flakes increased, hardness properties of the alloys increased. The specimens having high aspect ratio AlB_2 flakes have greater hardness values than lower ones.

Compared to the effect of the heat treatment on the hardness properties of the Al-B alloys, an additional of 14.5% increment was acquired on the hardness in comparison with non- heat treated sample.

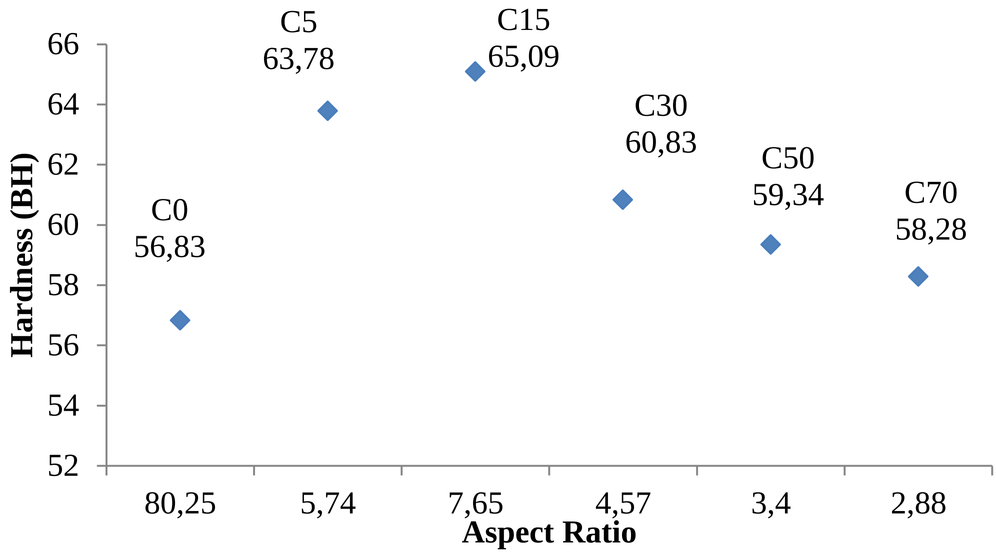


Figure 5.8 Relationships Between Hardness and Aspect Ratio of AlB_2 Flakes

5.5.2 Tensile Test Results

Tensile test is widely used to learn the basic design information about strength of materials. In this study, three cylindrical specimens for each condition were prepared according to TS 138 standard to conduct the test. Samples were produced and shaped with a diameter of 15 mm and gauge length 75 mm. Tensile test results were recorded depending on holding time in the furnace. Results were compared with heat treated Al-B alloy samples and pure aluminium casted at 1000^{o}C for 45 minutes.

Table 5.6 Tensile Test Results

Sample	Meas.1 (MPa)	Meas.2 (MPa)	Meas.3 (MPa)	Ave. (MPa)
AA 1070	61.6	57.5	59.9	59.67
C0	76.1	77.8	80.5	78.13
C5	87.4	84.9	85.3	85.87
C15	89.3	92.1	90.7	90.70
C30	79.4	80.3	81.6	80.43
C50	76.9	77.6	77.1	77.20
C70	75.9	73.4	76.2	75.17

As a result of tension test results shown in Table 5.6 and Figure 5.9, maximum ultimate tensile strength value of 90.7 MPa was measured from sample C15 held during 15 hours in the furnace and minimum ultimate tensile strength measurement of 75.17 MPa was recorded from sample C70 held during 70 hours in the furnace.

According to average tensile tests results, when holding time of heat treatment is increased, tensile strength of alloys are increasing up to 15 hours. But after 15 hours, the strength value of sample C30 decreased to 80.43 MPa. Later measurements are also showing that an increase in holding time is decreasing the UTS values to 77.20 MPa and 75.17 MPa, respectively.

Relationship between tensile strength and aspect ratio of AlB_2 flakes is shown in Figure 5.9. 70 hours heat treated sample C70 has the minimum aspect ratio of 2.88 and also this sample has the lowest UTS value of 75.17 MPa.

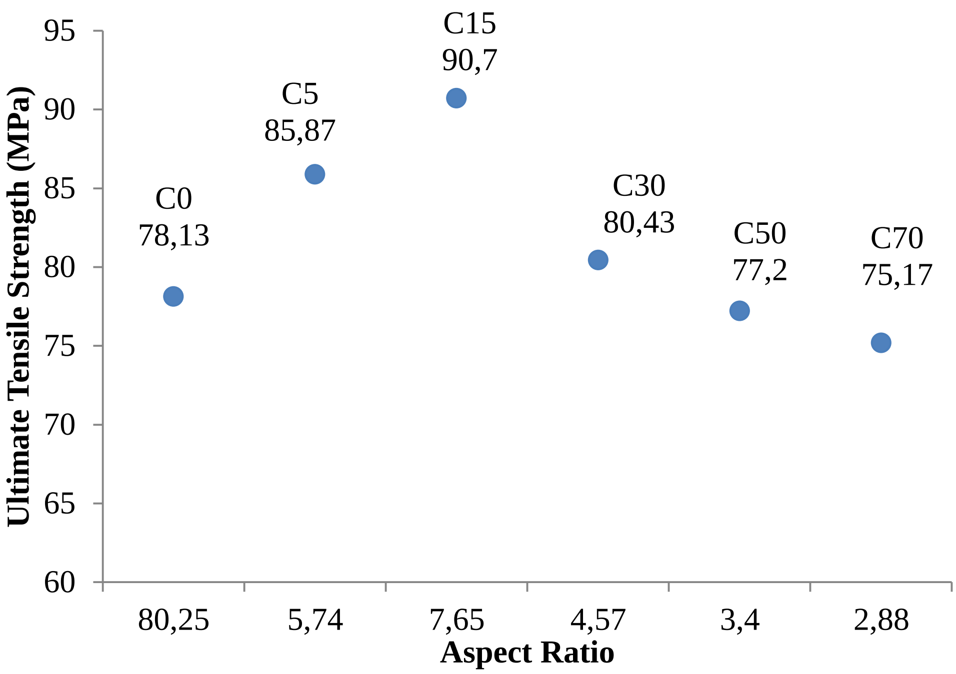


Figure 5.9 Relationships between Tensile Strength and Aspect Ratio of Flakes

Non-heat treated sample C0 has lower UTS than C15. As a result of comparison of the aspect ratio and UTS, it was shown that the specimens having high aspect ratio may have lower tensile strength values. However, as seen from Figure 5.7, AlB_2

flakes of C0 are very thinner and very long length than flakes of C15. This weakness causes a worsening effect on the tensile properties of sample C0.

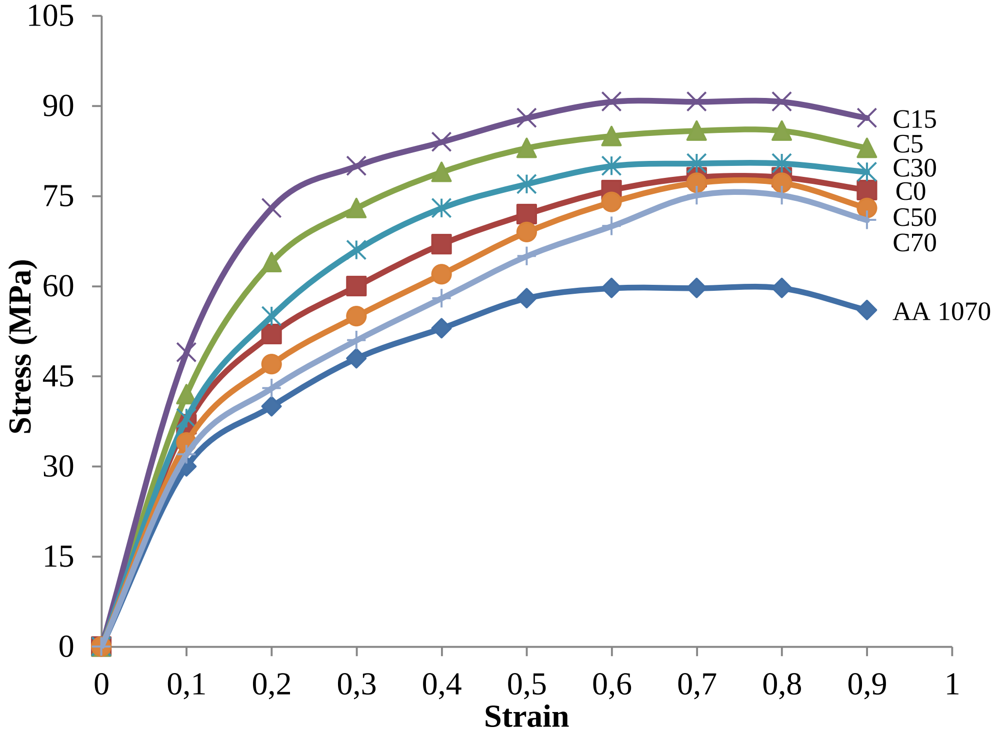


Figure 5.10 Stress-Strain Diagrams of Al-B Alloys

Addition of wt. 10% borax powders into pure aluminium is increasing the tensile strength values in a 31% increase with comparison with AA 1070. Heat treatment process is also changing tensile strength of Al-B alloys with respect to holding time at 550 ^{o}C in the furnace shown in Figure 5.10.

Tensile test results show that heat treatment has an increasing effect on the ultimate tensile strength of Al-B alloy up to holding 15 hours in the furnace in an additional 16% improvement compared with the non- heat treated sample. After greater holding time than 15 hours, tensile properties of the alloy are worsening. Depending on test results, heat treatment duration is playing a critical role on the tensile strength.

5.5.3 Impact Test Results

Impact tests are conducted to measure the resistance to failure of a material to a suddenly applied force. The test measures the impact energy or absorbed energy prior to fracture. The most common methods of measuring impact energy are Charpy and Izod impact energy tests. In this study, Charpy test method was applied to specimens having a length of 55 mm, a width of 10 mm and a height of 10 mm with a V-shaped notch, 2mm deep, with 45° angle and 0.25mm radius along the center of specimens.

Three specimens were prepared for each condition by using milling machine. Impact energy test results were recorded in Table 5.7. Change of impact energy depending on holding time and aspect ratio were compared with heat treated Al-B alloy samples and pure aluminium.

Table 5.7 Impact Test Results

Sample	**Meas.1 (kg.m)**	**Meas.2 (kg.m)**	**Meas.3 (kg.m)**	**Ave. (kg.m)**
AA 1070	5.4	5.9	5.2	5.50
C0	6.9	7.2	6.5	6.87
C5	7.6	7.1	6.8	7.17
C15	8.5	7.8	8.3	8.20
C30	5.6	6.4	5.9	5.97
C50	5.1	5.3	5.6	5.33
C70	5.8	4.2	4.8	4.93

From Table 5.7, it is seen that average impact energy measurement of 8.20 kg.m was recorded from sample C15 held with 15 hours in the furnace and minimum impact energy measurement of 4.93 kg.m was recorded from Al-B alloy C70 holded during 70 hours in the furnace.

According to average impact energy tests results, when holding time of heat treatment was increased, impact energy of alloys increased up to 15 hours. After longer holding time than 15 hours, impact energy value of sample C30 decreased to 5.97 kg.m. At greater holding times than 30 hours, impact energy values are worsening for C50, C70. There is a decrease on average values having 5.33 kg.m and 4.93 kg.m, respectively.

According to relationship between impact energy and aspect ratio of AlB_2 flakes is shown in Figure 5.11. Sample held 70 hours in the furnace, C70, has minimum aspect ratio of 2.88 and also this sample has the lowest impact energy value of 4.93 kg.m. Non-heat treated sample C0 has higher aspect ratio but it has lower impact energy value than 8.20 kg.m, strength value of sample C15.

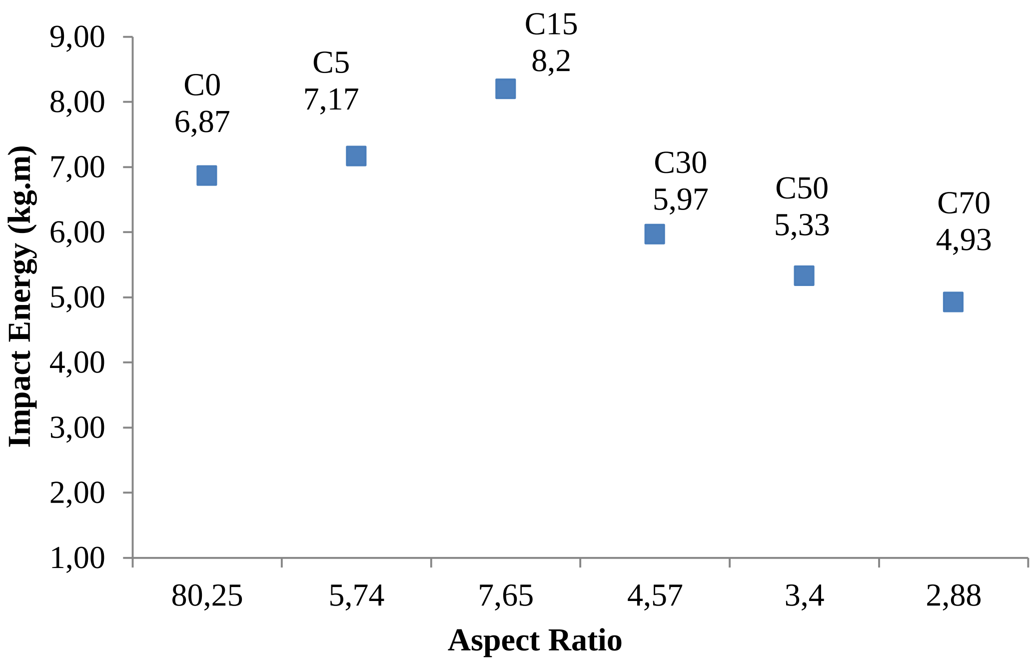


Figure 5.11 Relationship Between Impact Energy and Aspect Ratio of Flakes

It is thought that change of aspect ratio is an important parameter for the impact energy of Al-B alloys in this study. As a result of comparison the aspect ratio and impact energy, relationship between aspect ratio and impact energy had a linear change according to holding time in the furnace.

When aspect ratio of the flakes increased, impact energy of the alloys increased. In other words, Al-B alloys which had high aspect ratio AlB_2 flakes had higher impact energy values than lower ones.

Addition of wt. 10% Borax to pure aluminium, a change of 25% increment on the impact energy of Al-B alloy in comparison with pure aluminium. When looked at the effect of the heat treatment, heat treatment applied during 15 hours improved an additional 19% increment on impact energy compared with the non- heat treated sample.

5.6 PHYSICAL PROPERTIES

5.6.1 Electrical Conductivity Test

Electrical conductivity test was performed to compare the effect of the aspect ratio of AlB_2 flakes on the electrical conductivity of the Al-B alloys. Test results were presented in Figure 5.12.

It is seen that increasing holding time is getting better the conductivity of Al-B alloy up to holding 15 hours, maximum conductivity value of $3.79x10^7/\Omega m$ was obtained from sample C15.

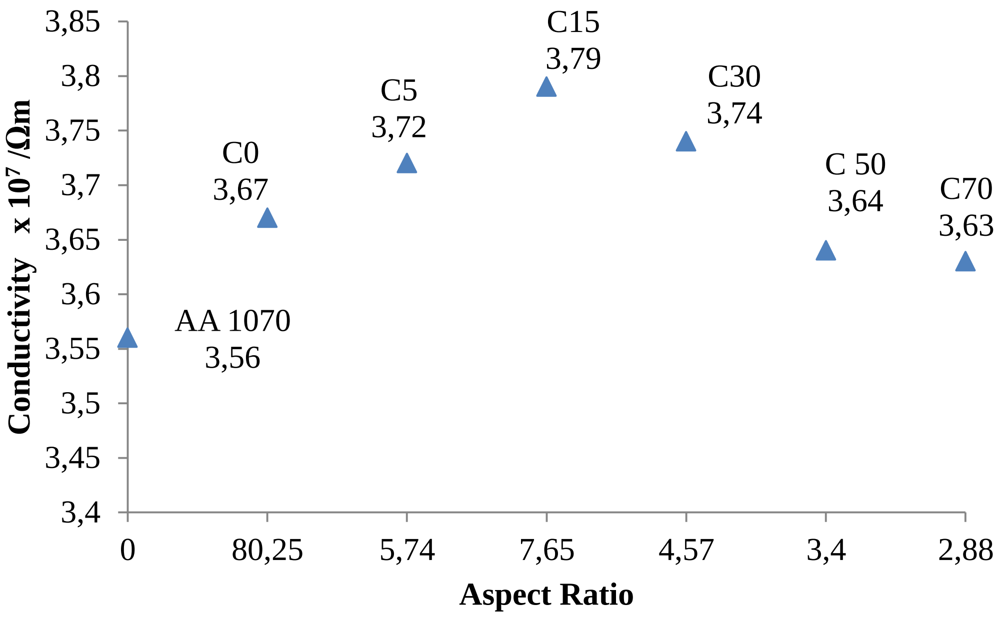


Figure 5.12 Change of Conductivity with respect to Aspect Ratio

After greater holding time more than 15 hours, the conductivity of C30 was decreased to $3.74x10^7/\Omega m$. During greater holding in the furnace more than 30 hours, the conductivity of C50, C70 is worsening and these average values are $3.64x10^7/\Omega m$ and $3.63x10^7/\Omega m$, respectively.

As a result of comparison the aspect ratio and conductivity, relationship between aspect ratio and conductivity had a parallel change based on holding time in the furnace.

Electrical conductivity and aspect ratio flakes of the alloy are increasing up to 15 hours holding time. Maximum conductivity and highest aspect ratio was measured from sample C15. After then, the conductivity results are starting to worsen. Sample 70 is presenting very poor conductivity and lowest aspect ratio particles.

According to microscopic examinations, it is thought that agglomeration of borides particles resulted in some disconnections between particles. And also flakes are transforming from rectangular flakes to circular flakes having low aspect ratio. Electrical conductivity of aluminium alloys varied depending on change of aspect ratio of the samples. An additional increase was obtained after heat treatment process with respect to non-heat treated sample, C0.

5.7 SCANNING ELECTRON MICROSCOPE ANALYSIS

In the end of the mechanical and physical tests and measurement of aspect ratio, sample C15 was greatest mechanical and physical properties than the other samples. Also microstructure of sample C15 is consisting of much more high aspect ratio AlB_2 flakes.

In order to display detailed shapes, characterization, reinforcement and dissolving of AlB_2 flakes in aluminium matrices, Scanning Electron Microscope (SEM) analyse was conducted.

As seen in Figure 5.13, clearly visible rectangular, plate and hexagonal shaped AlB_2 flakes are magnified and demonstrated. The boron particles were reinforced homogeneously in aluminium matrices.

A majority of the borides structures looks rectangular flakes morphology. Especially flakes having long length and low width and well-formed plate are reinforced in aluminium. There is no agglomeration between particles and generally flakes are independent and far from each other.

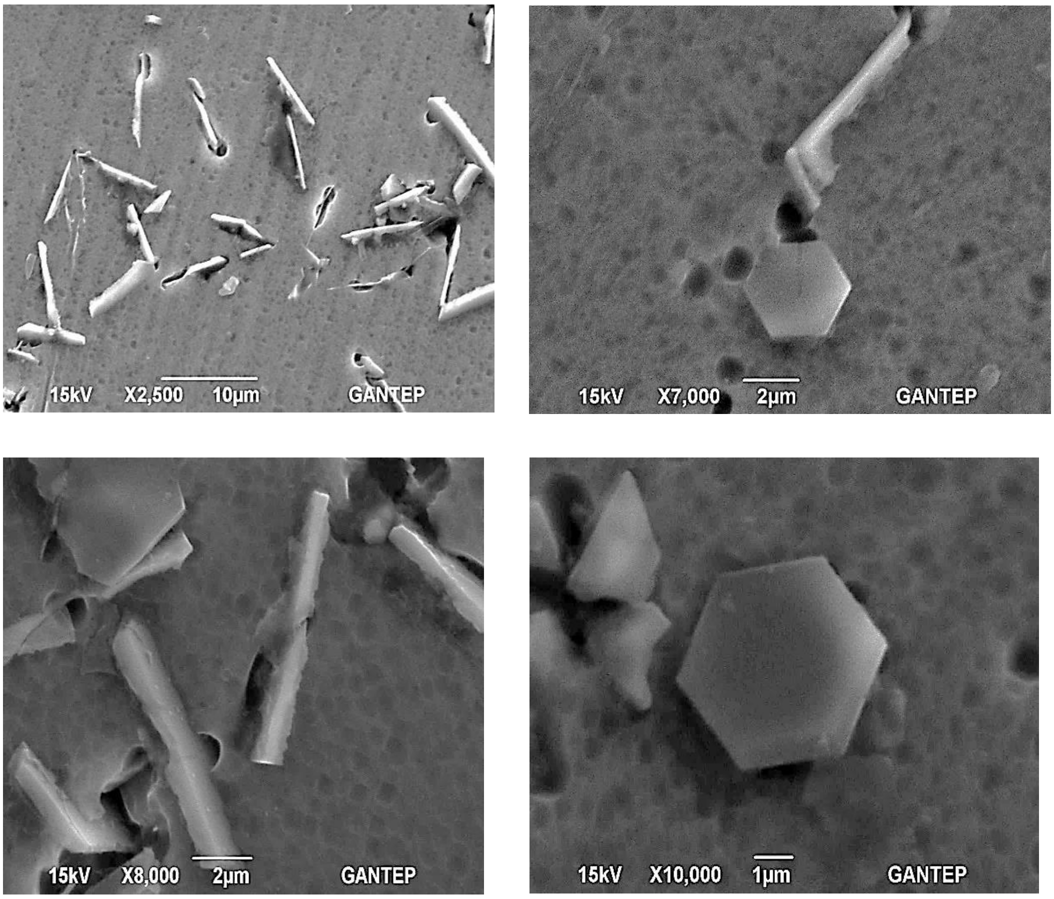


Figure 5.13 SEM Microstructure Pictures

5.8 DISCUSSION

Boron is an element which displays a peritectic reaction and has a partial solid solubility in aluminium matrices. When looking at Al-B phase diagram, liquid aluminium and AlB_{12} particles exist above 975°C. When temperature is decreased below 975°C, the formation AlB_2 particles begin.

The structures of AlB_{12} particles are unstable and fragile. Below peritectic temperature, thermodynamically more stable AlB_2 particles are formed. AlB_2 flakes have cylindrical, rectangular and hexagonal structure.

Aspect ratio of AlB_2 flakes in the matrix plays significantly role on the properties of the alloy. In this study, AlB_2 particles having different aspect ratios were fabricated in aluminium metal matrices.

It is realized that when aspect ratio of AlB_2 flakes increased, mechanical properties such as hardness, ultimate tensile strength, impact energy and physical properties such as electrical conductivity of alloys were increased by applying heat treatment processes to change aspect ratio of AlB_2 flakes. The transformation of low aspect ratio AlB_2 to high aspect ratio AlB_2 is achieved by execution of heat treatments.

Improved mechanical properties were obtained depending on transformation of low aspect ratio flakes to high aspect ratio AlB_2 flakes.

CHAPTER 6

CONCLUSIONS

In this thesis, fabrication and effect of aspect ratio on mechanical and physical properties of AlB_2 particles reinforced in aluminium metal matrix were studied.

Borax was used as a boron source due to its cheaper price and wide availability in the market. Borax powders wt. 10 % was added to molten aluminium holding at 1000°C in the furnace during 45 minutes.

Aluminium boron alloys were successfully fabricated by using casting operation. Mechanical tests such as hardness test, tension test, impact energy test and physical test such as electrical conductivity were performed to compare change of properties of alloys with respect to aspect ratio of AlB_2 flakes. Microstructural examinations were executed.

As a result of this study;

- AlB_2 particles were formed in aluminium matrices as a result of in-situ casting operation.

- Homogeneous distribution of boron particles was achieved by using casting process.

- Addition of borax in aluminium improved mechanical properties compared with pure aluminium.

- AlB_2 flakes have rectangular, cylindrical, hexagonal and spherical shaped structures. Cooling conditions are effective parameters on characterization, formation and shape of the AlB_2 flakes. Also it is thought that fast cooling rates such as air and water, provides formation of high aspect ratio flakes having long length and low width.

- Heat treatment process was executed to alloys in different holding times 5h, 15h, 30h, 50h and 70h, respectively in order to change length and width of flakes during experiments. As a result of microstructural examination, heat treatment is a very successful process to change length and width of the flakes.

- Average aspect ratio of AlB_2 flakes were measured and analysed by using software with optical microscope. Improved mechanical properties were obtained as a result of changing aspect ratio AlB_2 flakes. Heat treated samples more than 15 hours tends to worsen mechanical and physical properties. This shows that heat treatment duration is critical for mechanical and physical properties of Al-B alloys.

- It was observed that AlB_2 particles have higher aspect ratio when the holding time was increased to 15 hours. Presences of high aspect ratio AlB_2 in samples that had been obtained at 15 hours confirm that it had higher hardness, tensile, impact and electrical conductivity values due to high aspect ratio of boron particles. It is thought that long and flake-shaped particles strengthen mechanical and physical properties of Al-B alloys.

- Compared to the effect of the heat treatment on the hardness, a change of 14.5 % increase on the hardness in comparison with the non- heat treated sample.

- Tensile test results demonstrated that heat treatment provides an increasing effect on the ultimate tensile strength of Al-B alloy up to holding 15 hours in the furnace in an additional 16% improvement compared with the non- heat treated sample.

- An additional 19% increase on impact energy compared with the non- heat treated sample.

- According to microstructural pictures of the alloys, transformation of long flakes to circular form resulted in disconnections between particles and this confines displacements and movement of the electrons based on formation of low aspect ratio flakes. An additional improvement in electrical conductivity of alloy was obtained after heat treatment process with respect to non- heat treated sample based on having high aspect ratio flakes in the microstructure.

RECOMMENDATIONS FOR FUTURE STUDIES

Effect of aspect ratio AlB_2 flakes on mechanical and physical properties lacking in the Al-B alloys literature review was highlighted in this thesis. Future studies based on the aspect ratio of the Al-B flakes will guide usage of Al-B alloys in engineering applications and industry.

Following procedures, processes and methods will be lead and develop this study to one step further.

- Different alloying elements such as Mo, Cu, Ti can be reinforced to compare mechanical properties and physical properties Al-B alloy.

- B_2O_3, KBF_4 may be used as a boron source and the results can be compared and observed differences between yields.

- Studies may be concentrated on a special finished product.

- Effect of the production methods of Al-B alloys on aspect ratio of flakes can also be investigated.

REFERENCES

[1] Fridman Y. B. (1975). Mechanical properties of metals, *Metal Science and Heat Treatment*, **Vol. 17**, 637-637.

[2] ASM Specialty Handbook. (1993) Aluminum and Aluminum Alloys. ASM International Press

[3] Totten G. E., Mackenzie D. S. (2003). Handbook of Aluminium Alloy. Vol.2: Production and Materials Manufacturing. New York: Marcel Dekkel Inc.

[4] Ozturk M. (2011). An Experimental Study on Mechanical Properties of Aluminium-Boron Compounds. M.Sc. Thesis in Mechanical Engineering, Gaziantep.

[5] Everett R. K., Arsenault R. J. (1991). Metal matrix composites: Processing and Interfaces New York: Akademic Press.

[6] Daniel B. S., Murthy V. S., Murty G. S. (1997). Metal-ceramic composites via in-situ methods, *Journal of Materials Process and Technology*, **68**, 132-155.

[7] Ramana R. T. V. (2003). Metal Casting: Principles and Practice. New Delhi: New Age International Publisher.

[8] Davim J. P. (2014). Metal Matrix Composites: Materials, Manufacturing and Engineering. Berlin: CPI Books GmbH.

[9] Cooper P. S., Kearns M. A. (1996). Removal of transition metal impurities in aluminium melts by boron additives, *Materials Science Forum*, **Vol. 217-222**, 141-146.

[10] Funk H. Z. (1925). *Zeitschrift für Anorganische und Allgemenie Chemie*, **142**,269.

[11] Savas O. ,Kayikci R. (2013). A Taguchi optimisation for production of Al-B master alloys using boron oxide, *Journal of Alloys and Compounds ,**580,*** 232-238.

[12] Deppisch C., Liu G., Hall A. (1998). The crystallization and growth of AlB2 single crystal flakes in aluminium, *Journal of Materials Research*, **13(12)**, 3485−3498.

[13] Hall A. C., Economy J. (2000). Preparing high and low aspect ratio AlB_2 flakes from borax or boron oxide, *The Journal of The Minerals, Metals & Materials*, **52**, 42–44.

[14] Wang X. (2005). The formation of AlB_2 in an Al-B master alloy, *Journal of Alloys and Compounds*, **403**, 283-287.

[15] Wang Q. L., Zhao H. S., Li Z. G., Shen L., Zhao J. Z. (2013). Production of Al_2B master alloys by mixing KBF_4 salt into molten aluminium, *Transactions of Nonferrous Metals Society of China*, 294-300.

[16] Dragut D. V., Moldovan P., Butu M., Usurelu E. (2011). Characterization of in-situ AA6060/AlB_2 metal matrix composite, *UPB Scientific Bulletin, Series B: Chemistry and Materials Science,***Vol. 73,** 161-170.

[17] Birol Y. (2009). Production of Al−B alloy by heating Al/KBF_4 powder blends, *Journal of Alloys and Compound*, **481**, 195−198.

[18] Birol Y. (2013). Response to thermal exposure of ball-milled aluminium-borax powder blends, *Metallurgical and Materials Transactions*, **Vol. 44B**, 359−364.

[19] Birol Y. (2012). Aluminothermic reduction of boron oxide for the manufacture of Al-B alloys, *Materials Chemistry and Physics*, **136**, 963−966.

[20] Kayikci R., Kurtulus O., Gurbuz R. (2009). The formation and growth behaviour of aluminium boride crystals in an Al-B alloy, *Solid State Phenomena*, **Vol. 144**, 140-144.

[21] Savas O., Kayikci R., Koksal S. (2012). Application of Taguchi method to investigate the effect of some factors on in-situ formed flake structures of Al/AlB_2 composite, *Advanced Composite Letters,* **Vol.21**, 44-50.

[22] Kayikci R., Savas O. (2014). Fabrication and properties of functionally graded Al/AlB_2 composites, *Journal of Composite Materials,* 1-9.

[23] Zhu N., Jun G., Liang L. and Zhang D. (2007). Production of boron carbide reinforced 2024 aluminum matrix composites by mechanical alloying, *Materials Transactions*, **Vol. 48,**990-995.

[24] Deppisch C., Liu G., Shang J. K., Economy J. (1997). Processing and mechanical properties of AlB_2 flake reinforced Al-alloy composites, *Materials Science and Engineering*, **A225**, 153−161.

[25] Khatami N., Mirdamadi S. (2014). Effect of controlled quenching on the aging of 2024 aluminium alloy containing boron, *Journal of Materials Science &Engineering*,**Vol. 11**,17-26.

[26] Yilmaz N. F., Ozturk M. (2012). Improvement of mechanical properties of AA1070 aluminium by the addition of borax under different holding conditions, *Materialwissenschaft und Werkstofftechnik,* **43, No. 12**, 1006-1018.

[27] Available at: http://www.aluminiumdesign.net/why-aluminium/properties-of-aluminium/ (10.07.2015)

[28] American Society for Metals. (1971). Aluminium: Properties, physical metallurgy and phase diagrams. Ohio: Chapman & Hall Ltd.

[29] Available at: http://en.etimaden.gov.tr/about-boron-62s.htm (10.07.2015)

[30] Available at: http://www.boren.gov.tr/en/boron/boron-element (10.07.2015)

[31] Available at: http://www.boren.gov.tr/en/boron/areas-of-application/health (10.07.2015)

[32] Herring H. W. (1966). Selected mechanical and physical properties of boron Filaments. NASA Langley Research Center, Hampton, VA, United States.

[33] Eti Mine Works General Management. (2010). Eti Mine Works 2009 Annual Report. Ankara

[34] Rogathi P. K., Ray S., Liu Y. (1988). ASM Handbook, Vol 18, Ohio.

[35] Stefanescu D.M. (1993). Key Engineering Materials, Vol.79-80 Trans. Tech. Pub. Switzerland.

[36] Bhaskar C. K., Jatinder K., Hari S. (2014). Production Technologies of Metal Matrix Composite: A Review, *International Journal of Research in Mechanical Engineering & Technology*, **Vol. 4**, 27-32.

[37] Hoseini M., Meratian M. (2005). Tensile properties of in-situ aluminium-alumina composites, *Material Letters*, **59,** 3414-3418.

[38] Srivatsan T. S., Ibrahim I. A., Mohamed F.A., Lavernia E.J. (1991). Processing techniques for particulate-reinforced metal aluminum matrix composites, *Journal of Materials Science*, **vol. 26**, 5965-5978.

[39] Sannino A. P., Rack H. J. (1995). Wear, 189, 1.

[40] Wohrer L. C., Wosilait A. A. (1977). Reinforcing Properties of AlB_2 Flakes, *Boron and Refractory Borides*, 565-580.

[41] Sigworth G. K. (1984). Grain refining of aluminium and phase relationships in the Al-Ti-B system, *Physical Metallurgy and Materials Science*, **Vol.15A**, 277-282.

[42] Handbook of Ternary Alloy Phase Diagram. (1995). ASTM.

[43] Hall A. C., Economy J. (2000). The $Al+AlB_{12}\leftrightarrow AlB_2$ peritectic transformation and its role in the formation of high aspect ratio AlB_2 flakes, *Journal of Phase Equilibrium*, **21**, 63–69.

[44] International Aluminium Institute. (http://www.world-aluminium.org) (07.09.2015)

[45] Duque N. B., Melgarejo Z. H., Suarez O. M. (2005). “Functionally graded aluminium matrix composites produced by centrifugal casting”, *Materials Characterization*, **55**, 167– 171.