An Experimental Study on Mechanical Properties of Aluminum-Boron Compounds

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ABSTRACT

AN EXPERIMENTAL STUDY ON MECHANICAL PROPERTIES OF ALUMINUM-BORON COMPOUNDS

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In this thesis, mechanical properties of Al-B alloys, prepared by the addition of different amounts of borax to 99.70% pure commercial aluminum, were investigated. In this preparation process, amount of borax, holding temperature and holding time were realized as affecting parameters. Additive amount of borax were selected to be 5, 10, 15 and 20 percentage while holding temperatures were 800, 1000 and 1200^0C and holding times were 30, 45 and 60 minutes before casting into dies. By considering these parameters three specimens were prepared for each condition. These specimens were then subjected to mechanical tests such as tensile, impact and hardness tests and average values were recorded. As a result of mechanical tests, tensile strength of the alloy with the incorporation of borax was found to increase by about 33% in comparison to the pure aluminum without borax while 21% increase in impact energy and 54% in hardness value. A substantial quantity of aluminum-boride flakes were observed in the microstructural investigation of the samples. Microscopic observations of alloyed samples reveal homogeneous distribution of aluminum boride particles and fewer porosity levels. It is observed that mechanical properties of AA 1070 aluminum can be improved by incorporating with borax.

Key Words: Boron Compounds, Borax, Al-B alloy, Mechanical Properties

ÖZET

ALUMİNYUM BOR BİLEŞİKLERİNİN MEKANİK ÖZELLİKLERİ ÜZERİNE DENEYSEL BİR ÇALIŞMA

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Bu tezde, 99.70% saflıktaki ticari alüminyuma farklı oranlarda borax katılarak oluşturulan Al-B alaşımlarının mekanik özellikleri incelenmiştir. Deneyler süresince, borax miktarı, bekletme sıcaklığı ve bekletme süresi gibi parametreler kullanılmıştır. Üretim aşamasında borax ilave oranları yüzde 5, 10, 15 20 olarak seçilirken, bekletme sıcaklıkları 800, 1000 ve 1200^{0}C ve bekletme süreleri de kalıba dökmeden önce 30, 45 ve 60 dakika olarak belirlenmiştir. Bu parametreler uygulanarak her koşul için 3'er tane numune üretilmiştir. Üretilen numuneler çekme, darbe ve sertlik testi gibi mekanik testlere tabi tutulmuş ve çıkan sonuçların ortalama değerleri alınarak kaydedilmiştir. Mekanik testlerin sonucunda içine borax katılarak oluşturulan alaşımın saf alüminyuma göre çekme mukavemetinde 33%, darbe mukavemetinde 21% ve sertlik ölçümlerinde de 54% artış gözlemlenmiştir. Numunelerin mikroyapıları incelendiğinde çok sayıda alüminyum-borur parçacıklarına rastlanmıştır. Mikroskobik incelemeler alüminyum-borur parçacıklarının alüminyum içinde homojen bir şekilde dağıldığını ve gözenek seviyesinin düşük olduğunu ortaya koymaktadır. AA 1070 alüminyumuna borax eklenmesi ile alaşım oluşturularak mekanik özelliklerinin iyileştirilebileceği görülmüştür.

Anahtar Kelimeler: Bor Bileşenleri, Borax, Al-B alaşımları, Mekanik Özellikler

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CHAPTER 1

INTRODUCTION

In recent years, many research activities have been observed on aluminum alloys. Al alloys are often used in aerospace, aircraft, military, transportation, sport fields and energy sectors. The most important characteristics of Al alloys are low density, high strength-weight ratio and resistance to corrosion that have enabled them to be one of the dominant structural material families of the 21th century.

Aluminum alloys are alloys in which Al is the predominant metal. Al has low density (2.71 g/cm^3) compared with other engineering materials. It is nearly one-third as dense as steel; is malleable, ductile, and easily machined and cast; and has excellent corrosion resistance because of the continuous, protective oxide film that forms very quickly in the air, and good workability that enables Al and its alloys to be economically rolled, extruded, or forged into useful shapes [1].

The mechanical properties of Al can be greatly improved by incorporating with some chemical elements. Pure aluminum has a tensile strength of about 49 MPa and if it is formed into an alloy with a suitable heat treatment this value can be increased up to around 700 MPa.

Most of the chemical elements possessing metallic characteristics readily alloy with aluminum, but only a few are important major alloying ingredients in commercial aluminum-base alloys. Nevertheless, an appreciable number of other elements serve as supplementary alloying additions for improving alloy properties and characteristics. The typical alloying elements are copper, magnesium, manganese, silicon, and zinc [2].

In this research, boron element was used as an alloying element to investigate a new generation aluminum alloy. Boron was selected because of strategic and primary importance for Turkey. Turkey holds the largest boron deposits in the world that is estimated to be %64 of proved boron reserves and also is sufficient to meet current global boron demand around 400 years alone. In terms of its reserve potential, variety of boron minerals, tenor quality of the deposits and low extraction costs, boron minerals have implied for Turkey to be one of the promising sources of economic development for long years [3].

Boron forms compounds with several metal and non-metal elements and these compounds exhibit different properties. Boron minerals, borates and compounds have very wide range of applications in industry, such as agriculture, detergents and soaps, ceramic glazes and enamel frits, insulation fiberglass, flame retardants, additive for solid rocket propellants, nuclear power stations and so on.

In this study, the aim is to examine the mechanical properties of Al-B alloys. Conventional casting method was used to produce aluminum-boron alloys. 99.70% pure commercial aluminum and borax powder were used as the alloy materials. Boron is reduced from borax by aluminum and dispersed into the aluminum melt in the forms of aluminum borides. Several parameters were taken into consideration; such as borax addition, holding temperature and holding time. Borax addition was performed within 5, 10, 15 and 20 weight percentage of pure aluminum and three temperatures, namely: 800, 1000 and 1200^0C were carried out during casting process. All samples were also hold 30, 45 and 60 minutes in the oven at elevated temperatures before casting into dies.

All samples were subjected to tensile test, impact test and hardness test to observe the variations of mechanical properties. And also metallographic studies were performed to observe the microstructural characterization of the Al-B alloys.

The related literature search describing previous investigations on the fabrication and properties of Al-B alloys are summarized in Chapter 2.

It is noticed that there were serious gaps in the literature concerning the mechanical properties of Al-B alloys. Hence detailed studies were carried out in this area.

In Chapter 3, the general knowledge on aluminum and borax are presented. General properties and application areas of these materials are given in this chapter.

In Chapter 4, the production steps of Al-B alloys are described. Experimental setup, applied mechanical tests and metallographic studies on the samples were explained briefly.

In Chapter 5, experimental results obtained from different mechanical tests are outlined according to the parameters. Microscopic investigations are also given in this chapter.

In chapter 6, highlights of this study and achieved results are concluded. Recommendations for future studies are also given in this chapter.

CHAPTER 2

LITERATURE SURVEY

2.1 INTRODUCTION

In this chapter the most relevant studies about aluminum and boron compounds are outlined. Since wide application areas of boron and excellent physical and mechanical properties of aluminum, there are some studies on aluminum and boron materials as an alloy and composite.

2.2 LITERATURE ON ALUMINUM-BORON ALLOY

Increasing worldwide interest in the alloy and composite materials leads to researchers to study on enhancing of new materials to improve physical and mechanical properties of existing materials.

An experimental study on the formation of AlB_2 in an Al-B master alloy was carried out by Wang [4]. In this study two types of boride phases in an Al-3 wt. % B master alloy were produced by chemical reactions of aluminum and KBF_4. By the investigation of XRD in SEM, the two types of boride phases were distinguished as AlB_2 on the surface of AlB_{12}. He explained the presence of AlB_{12} inside AlB_2 particles in the Al-3 wt. % B master alloy was attributed to the sluggish nature of the peritectic reaction between AlB_{12} and aluminum.

The crystallization and growth of AlB_2 single crystal flakes in aluminum were investigated by Deppisch et al [5]. An in situ high temperature heat treatment was used to investigate the crystallization and growth behavior of AlB_2 flakes in aluminum. In their experiments aluminum samples containing 1.8% boron were heated above the liquidus and then rapidly cooled through the Al(L)+AlB_{12} region to avoid the formation of AlB_{12} crystals. They imposed various parameters on the samples during their experiments such as hold time, temperature, and cooling rate. When hold time increased in the Al(L)+AlB_2 region, flakes were yielded thicker and wider while decreasing the flake cross-sectional density and growth rate increased at elevated hold temperatures in the same region. And also the microstructure of the composites was significantly affected by varying the cooling rate in the high temperature quenching experiments from above the liquidus temperature to room temperature. The data revealed that by increasing the cooling rate the average flake thickness decreased while the average flake width increased.

Yücel [6] described an improved practice to manufacture Al-Ti-B master alloys by reacting halide salts with molten aluminum. In his experimental studies, 99.7% pure aluminum ingot was used with K_2TiF_6 and KBF_4 salts to produce master alloy. He employed a well established "halide salt" route to produce Al-Ti-B grain refiner alloys with consistent, good properties. In the study, holding step in the production cycle was revised, however, to avoid oxidation of the molten alloy. Holding temperatures higher than 800^0C and holding times longer than 30 minutes both had an undesirable effect on the grain refining performance. Also stirring during holding was found to degrade the grain refining properties.

Yücel [7] also used aluminum powder instead of ingot to produce Al-B alloy. In this study, Al and KBF_4 powder blend samples prepared by mixing and ball milling. When these samples were heated, KBF_4 started to react with aluminum at 490˚C, well below the melting point of aluminum. AlB_2 particles were dispersed more homogeneously in ball milled samples and no evidence of the AlB_{12} phase, often reported to be present in Al-B master alloys, was noted in heat treated powder blend samples.

Yücel [8, 9] also performed an experimental study to replace the KBF_4 salt with B_2O_3 and $Na_2B_4O_7$ to reduce the volume of fluoride salts to be added to molten aluminum in the production of Al-Ti-B grain refiners. Replacing KBF_4 salt with B_2O_3 impaired the grain refining performance and with the dross generated, hurt the fluidity of the melt making pouring difficult. A significant improvement was noted in the Ti recovery and in the microstructural features when B_2O_3 was added with a drossing flux to promote the separation of oxides from the molten alloy. His experiments showed that the best result was obtained when B_2O_3 and KBF_4 were used together to supply boron. On the other hand the Al-Ti-B grain refiner alloy which relied on $Na_2B_4O_7$ for the boron supply, offered an acceptable grain refining performance in spite of a relatively lower Ti recovery. This performance came, however, with a slight decrease in the alloy recovery which can be readily compensated with additional particulate charge. According to his experimental results it is fair to conclude that $Na_2B_4O_7$ is a viable alternative for KBF_4 in the popular halide salt route.

Tee et al [10] studied the mechanical properties of in-situ Al-TiB_2 composite by incorporation of carbon. In-situ TiB_2 reinforced aluminum composites was successfully synthesized through chemical reactions between elemental Al, B and Ti materials. By the incorporation of carbon into the in-situ reaction, Al_3Ti intermetallic phase was found to decrease via the formation of TiC and the decomposition of Al_3Ti since TiC is more stable in the presence of carbon. In this study mechanical properties in terms of yield and ultimate tensile stresses and Young's modulus were improved. Ductility, in terms of elongation of the composite with the incorporation of carbon, was found to increase by about 26% in comparison to the composite without carbon.

An experimental study of the fabrication and properties of boron carbide-reinforced aluminum 1100 matrix composites were carried out by Mohanty et al [11]. In this study aluminum-boron carbide composites up to 25 wt% boron carbide (B_4C) particulate reinforcement in aluminum 1100 matrix were developed at 600^0C and the resulting microstructure and physico-mechanical properties were studied.

Experimental investigation and thermodynamic assessment of the Al-rich side of the Al-B system was performed by Fjellstedt et al [12]. They analysed the phase relations in the Al-Ti-B system for the manufacturing of Al-TiB_2 composites. In their studies, the eutectic temperature was found to agree well with the literature. However, the peritectic temperature was found to be 914^0C above the liquidus line.

Fjellstedt et al [13] presented an experimental analysis of the intermediary phases AlB_2, AlB_{12} and TiB_2 in the Al-B and Al-Ti-B systems. They studied the phases in

Al-B and Al-Ti-B alloys produced by several different sample fabrication methods. Samples were examined using light optical microscopy, energy dispersive X-ray spectroscopic analysis (EDS) and X-ray diffraction analysis (XRD). The results indicated that only a limited solubility of Al in TiB_2 and Ti in AlB_2 exists, hence a continuous compound $(Al,Ti)B_2$ is not stable.

Hall and Economy [14] performed a study based on a preparing high and low aspect ratio AlB_2 flakes from borax or boron oxide. They developed a new synthesis that allows AlB_2 to be prepared directly from the reaction of borax ($Na_2B_4O_7 \cdot 10H_2O$) or boron oxide (B_2O_3) with aluminum. Aluminum at temperatures higher than 900˚C was shown to reduce these boron containing compounds, producing an Al_2O_3 containing slag and AlB_2. A natural separation occurs, leaving AlB_2 in the molten aluminum and Al_2O_3 as part of a slag that forms at the melt surface.

Economy et al [15] developed a new family of metal matrix composites (MMCs) based on high aspect ratio AlB_2. They conversed the low aspect ratio AlB_2 to high aspect ratio AlB_2 when preparing these MMCs. This conversion was associated with the $Al(L)+AlB_{12} \leftrightarrow AlB_2$ peritectic transformation. Differential scanning calorimetry and optical microscopy was used to investigate this transformation. In this study, the equilibrium Al-B peritectic temperature was determined as 956±5˚C.

The effect of holding conditions in the conventional halide salt process on the performance of Al-Ti-B grain refiner alloys has been put into perspective by Yücel [16]. According to his experiments holding the molten alloy at 800^0C for 30 min under induction stirring in the conventional halide salt method gives the best performance. Higher temperatures, longer holding times and mechanical, instead of induction, stirring during holding lead to an extensive oxidation of the melt. On the other hand short holding times leave the salt reaction incomplete. The impact of the salt addition practice on the microstructure and grain refining efficiency of Al-Ti-B alloys produced by the "halide salt" route was also investigated by Yücel [17]. He optimized the grain refining performance of an experimental Al-5Ti-B master alloy due to premix the halide salts before addition to molten aluminum at 800^0C during the production of the master alloy. In view of his experiments, an improved salt addition practice to ensure an exceptional grain refining performance is claimed to comprise the following steps: melting commercial purity aluminum ingot; addition of pre-mixed salts to molten aluminum at 800^0C, at once to facilitate a rapid salt reaction, gently mixing the salts with the aluminum melt without introducing any stirring.

Halverson et al [18] studied on processing of boron carbide-aluminum composites. They established a reliable criterion for fabricating of B_4C-Al particulate composites. In these materials, chemical reactions occur between 800^0C and 1400^0C. Since chemical reactions cannot be eliminated, they processed B_4C-Al by rapidly heating to near 1200^0C to ensure wetting of B_4C by molten aluminum and subsequently heat-treating below 1200^0C for microstructural development. Also pressure was applied to accelerate densification faster than the kinetics of phase formation. On the other hand, Arslan and Kalemtas [19] studied the same compositions with adding silicon carbide. The aim of their study was to shed light on the wetting mechanism in the SiC-B_4C-Al system to explore processing routes that enable infiltration of Al alloys into these ceramic powder mixtures without the formation of the deleterious reaction product AlC3.

Microstructural variations in cast B_4C-reinforced aluminum matrix composites were discussed by Kerti and Toptan [20]. Their work aimed to introduce a cost effective and reliable casting technique to overcome the wetting problem between B_4C and liquid aluminum metal as well as the formation of undesirable phases at the interface using K_2TiF_6 flux. For this aim, experimental work was carried out by using B_4C powders with different particle sizes to reinforce commercially available aluminum using casting technique. As a result of their experiments B_4C addition with bigger particle size resulted in better microstructure with free of agglomerated particles.

Zhang et al [21] described particle distribution and interfacial reactions of Al-Si- B_4C die casting composites. In their study, attention was particularly focused on the particle distribution and interface reaction products between B_4C particles and the aluminum matrix. It was shown that the B_4C particle distribution in the cross-section of the cast part was not uniform in their samples. It was found that the barrier layer consists of three sublayers; Al_3BC, TiB_2, and Si between the B_4C particles and aluminum.

Muktepavela et al [22] studied on the structure and mechanical properties of Al-B composite powder. They investigated structural peculiarities and microhardness of separate powder particles (d≈1mm) which was obtained by crushing pieces of composite material presenting industrial waste. As a result of their investigation the powder represented a new structurally non-homogenous material with the increased microhardness in near surface layers. Also stable oxide compounds were formed on internal surfaces and defects of the aluminum alloy.

Effect of the boron content in the aluminum-boron composite was presented by Abenojar et al [23]. They used powder isostatic compaction and sintering as consolidation method. Aluminum-boron metal matrix composites were fabricated and tested changing the boron content (10-50 wt. %). Depending on their analysis low boron (10-20 wt. %) materials, where aluminum was the predominant phase, cannot be sintered at temperatures over aluminum melting point. Bending strength of these materials was in all cases worse than the pure aluminum ones and decreasing with the addition of boron. High sintering temperature improved properties, and composites presented higher hardness than aluminum.

2.3 CONCLUSION OF LITERATURE REVIEW

According to the literature survey it is realized that almost all studies are concentrated on microstructural variations. The most of the emphasis is given to grain refinement, phase formation and transition. However there is less work on the mechanical properties of Al-B alloys. Therefore, this thesis attempts to fill this gap on the effect of boron content in the alloy in reference to mechanical properties.

CHAPTER 3

PROPERTIES AND IMPORTANCE OF ALUMINUM AND BORON

3.1 INTRODUCTION

The aim of this chapter is to present general knowledge on boron and aluminum materials. Information about history of boron, physical and chemical properties of boron and aluminum and the use of boron and aluminum fields in industry are explained briefly in this chapter.

3.2 BORON

The elemental boron (B) is widely distributed in nature. Because of its high affinity for oxygen, boron does not occur in nature as a free element. There are over 200 naturally occurring boron containing minerals. Apart from their occurrence in a few commercially exploitable deposits (mainly as sodium or calcium borate minerals), borates are present everywhere at low concentrations, in rocks, soils, fresh waters and sea water [24]. Commercially important ones are tincal, colemanite, ulexite and kernite. These ores are transformed into boron derivative products by refining and chemical processing. Industrially, pure isolated boron is produced with difficulty, as boron tends to form refractory materials containing small amounts of carbon or other elements. Boron products are used in close to 500 different areas including space and aeronautics, nuclear applications, fuels, agriculture, glass industry and chemicals and detergents etc. Boron compounds play specialized roles as high-strength lightweight structural and refractory materials.

3.2.1 History of Boron

Boron compounds have been known for thousands of years. The Babylonians have been credited with importing borax from the Far East over 4000 years ago for use as a flux for working gold. Mummifying, medicinal and metallurgic applications of boron are sometimes attributed to the ancient Egyptians. None of this very old borax history has been verified, but solid evidence exists that tincal (i.e., $Na_2B_40_7$-$10H_20$, the mineral borax) was first used in the 8th century around Mecca and Medina, had been brought there (and to China) by Arab traders. The earliest source of borax is believed to have been Tibetan lakes. The borax was transported in bags tied to sheep, which were driven over the Himalayas to India. The use of borax flux by European goldsmiths dates to about the 12th century [25].

The element was isolated in 1808 by Sir Humphry Davy, Joeseph Louis Gay-Lussac, and Lois Jacques Thenard, to about 50 percent purity. These scientists did not recognize the substance as an element. Jöns Jakob Berzelius identified boron as an element in 1824. The first pure boron was produced by the American chemist W. Weintraub in 1909 [24].

3.2.2 Importance of Boron for Turkey

Technological developments in the world increase the importance and use of boron rapidly. This situation can be considered as an important advantage if Turkey uses the production, consumption and marketing strategies effectively. In terms of its reserve potential, variety of boron minerals, tenor quality of the deposits and low extraction costs, boron minerals have implied for Turkey to be one of the promising sources of economic development for long years.

Turkey has a strategic position due to its geographical features and wide variety of mines. Among the mines; Boron is undoubtedly the most remarkable one that may make Turkey the leading country in the market according to its reserve and producing capacity. Turkey holds the largest boron reserves in the world that is estimated to be %67 of known boron reserves and also is sufficient to meet current global boron demand around 400 years alone [27].

When considered as reserves based on B_2O_3, total boron reserves of the world are 1.176 million tonnes. This includes 369 thousand tonnes of definite and 807 thousand tonnes of probable or possible reserve. With respect to the boron reserves of Turkey, they are 851 thousand tonnes which includes 227 thousand tonnes of definite and 624 thousand tonnes of probable or possible reserve. In addition to this, Turkey's share of total world boron reserves is 72% as reserves based on B_2O_3 (Table 3.1).

Table 3.1 World Boron Reserves (Thousand Tons - B_2O_3) [27]

Country	Proven Reserve	Probable Possible Reserve	Total Reserve	Percent in Total (%)
Turkey	227.000	624.000	851.000	72.20
U.S.A.	40.000	40.000	80.000	6.80
Russia	40.000	60.000	100.000	8.50
China	27.000	9.000	36.000	3.10
Chile	8.000	33.000	41.000	3.50
Bolivia	4.000	15.000	19.000	1.60
Peru	4.000	18.000	22.000	1.90
Argentina	2.000	7.000	9.000	0.80
Kazakhstan	14.000	1.000	15.000	1.30
Serbia	3	0	3	0.30
Total	**369.000**	**807.000**	**1,176.000**	**100**

Tincal, colemanite and ulexite are known as important boron minerals of Turkey. Boron minerals contain different amount of B_2O_3 in their structures. The important factor for industrial application of boron minerals is B_2O_3 content. Boron deposits in Turkey are rich in B_2O_3. Processing of boron minerals in Turkey has been only executed by Eti Mine Works [28]. This foundation with five establishments set up in various regions, such as Eskisehir-Kirka, Balikesir-Bigadic, Balikesir-Bandırma, Bursa-Kestelek and Kutahya-Emet has produced the boron raw concentrate (Figure 3.1).

Figure 3.1 Turkey Boron Reserves [26]

The most outstanding countries producing boron are Turkey, USA, Argentina, Russia, China and Chili. In 2009 Turkey is the biggest borate producer of the world, which has produced 39 percent of the world while USA has produced 30 percent [27]. But when looking the reserve amounts of Turkey (72.2%) and USA (6.8%), Turkey has 12 times greater amounts of reserves than USA. What is striking about the issue is that, Turkey has not generated the expected benefits from its natural endowment in boron minerals, which are considered to be the one of the most important and valuable resources of Turkey.

3.2.3 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 gravities of 2.3 and 2.33 g/cm^3, respectively. Physical properties of boron are given in Table 3.2.

The physical properties of elemental boron are significantly affected by purity and crystal form. Boron is the second hardest element, the diamond allotrope of the carbon being the hardest [29]. All forms of boron have very high melting points, from 2200 to 2300°C. It has interesting optical characteristics, transmitting portions of the infrared lights, and is a poor conductor of electricity at room temperature, but a good conductor at high temperature. [31].

Table 3.2 Physical Properties of Boron [30]

Properties	Value
Atomic weight (Mass number)	10,81±0,005 (Number of Proton + Number of Neutron)
Melting point	2190±20°C
Boiling point	3660 °C
Density of boron(Crystalline 25-27°C)	2,33±0,002 g/cm^3
Density of boron(Amorphous 25-27°C)	2,3 g/cm^3
Heat capacity	1,54+0,0004 Tcal/g-atom-deg
Heat of combustion	306 ±1 kcal/g atom
Heat of vaporization	128 kcal/g atom
Heat of fusion	5.3 kcal/g atom
Knoop hardness	2100-2580 HK(kgf/mm^2)
Vickers hardness	5000 HV(kgf/mm^2)

Elemental boron (B) is a member of group III B of the periodic table. Atomic number and relative atomic mass number of boron are 5 and 10.81, respectively. The chemical behavior of elemental boron depends upon the morphology and particle size.

Generally speaking, crystaline boron is rather unreactive, whereas amorphous boron does react more readily. At room temperature all modifications of boron are relatively resistant to chemical attack. Reaction with oxygen starts at ≈ 600 ºC but is restrained by the formation of a glassy liquid B_2O_3 film on the boron particles [30].

One property of special importance is boron's ability to absorb neutrons. Neutrons are subatomic particles with no charge that occur in the nucleus of nearly all atoms. Boron atoms are able to absorb a large number of neutrons. This makes boron useful in the control rods of nuclear reactors.

3.2.4 Application Areas of Boron.

Boron products have numerous applications from medicine to nuclear. Major applications of boron include borosilicate glass, panel screen, reinforcement fibre glass, insulation fiber glass, detergent, agriculture, enamel, frits and ceramic glazes, pesticides, flame retardant, insecticides, timber and wood preservation. World Boron consumption by sectors is given in Figure 3.2 [27].

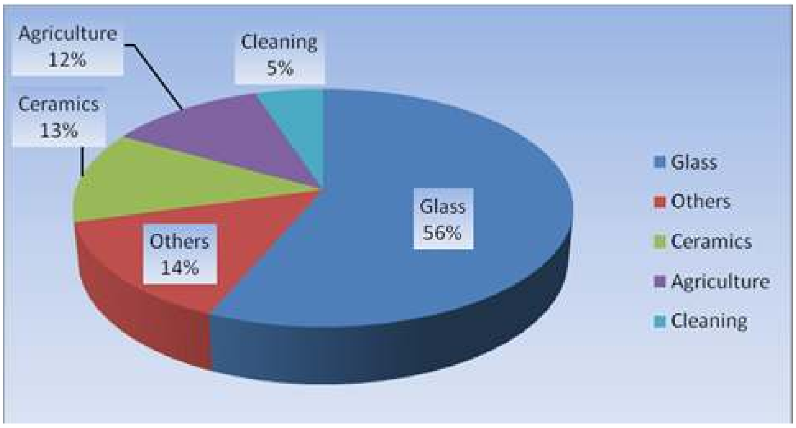

Figure 3.2 World Boron Consumption by Sectors [27]

Boron compounds may be classified under two groups with respect to their fabrication technologies and usage areas:

1) Compounds which are produced in large quantities: Boron mineral concentrates which are produced in large quantities with chemical engineering applications and widespread application areas, i.e. agriculture, glass production and detergents, etc.

2) Specialty boron chemicals that are produced in limited amounts and have special application areas. Elemental boron, boron fiber, boron carbide and similar specialty boron compounds require special production technologies and generally are used in high technology areas.

Elemental boron is used in very diverse industries from metallurgy to electronics. Other areas of application include ceramic, propulsion, pyrotechnics, and nuclear chemistry. Boron is nontoxic. Workplace hygienic practices, however, include avoiding the breathing of boron dust or fine powder [29].

Boron is used in pyrotechnics and flares to produce a green color. It has use in some rockets as an ignition source. Boron is also used in air bags, placed in automobiles as safety devices, for initiating the sodium azide (NaN_3) which fills the bag with nitrogen [29]. Dispersed mixture of boron and another metal as deoxidizing and degassing agents to harden steel, to increase the conductivity of copper in turbojet engines, and in making of brass and bronze. Two examples are alloys of ferroboron and magnese boron [29].

Cleaning and washing products use boron compounds. In North America, boron is mostly used as a washing aid and softener where ten percent of boron consumption is used in the cleaning industry. In Western Europe, sodium perborate is used as a bleaching agent in soap and detergent. Over 41% of their boron consumption is cleaning products [32].

Boron compounds are widely employed in many branches of the national economy, for example in medicine for the preparation of disinfectants and drugs, in the glass industry for the production of optic and chemical stable glass. [32].

Boron compounds are also considered as the most promising candidates among the metal hydrates for the safe storage of hydrogen which is an impediment to the commercial use of hydrogen as fuel in fuel cells. In recent years, scientific studies carried out on borohydrides have shown that borohydrides can be used commercially as hydrogen carrying material in fuel cells. Also, it is known that fuel cells technology converts hydrogen to electrical energy [28].

3.3 ALUMINUM

Aluminum is a soft and light weight metal with a dull silver appearance, due to a thin layer of oxidation that forms quickly when it is exposed to air. Aluminum is found primarily as the ore bauxite. Bauxite is mined then refined into aluminum oxide trihydrate (alumina) using the Bayer Process. Alumina is then electrolytically reduced via a smelting process into metallic aluminum.

Pure aluminum has a tensile strength of about 49 MPa and if it is formed into an alloy and with a suitable heat treatment this value can be reached to around 700 MPa [33]. Al is a low density metal that can be strengthened by the addition of alloying elements. Al is nearly one-third of steel density; is malleable, ductile, and easily machined and cast; and has excellent corrosion resistance and durability due to the protective oxide layer [1].

Since Al has high specific strength (strength/density), high electric conductivity, low density, high corrosion resistance and is cheaper than other light metals, the use of Al has been increasing rapidly [34]. The drawback of aluminum and its alloys is their low melting temperature (~660°C) that limits their maximum service temperature. However aluminum alloys and composites are widely used in aeronautical, military, automotive and sport fields due to not only some mechanical properties like high strength-weight ratio, elastic modulus, wear resistance, low density but also low thermal expansion coefficient, high electric conductivity and heat transfer coefficient [35].

3.3.1 Physical and Chemical Properties of Aluminum

Aluminum is the chemical element in the periodic table that has the symbol Al and atomic number 13. It is a silver and ductile member of the poor metal group of chemical elements. The most important physical property of Al is low density (2.71 g/cm^3) which is superior to aluminum from other engineering materials. Al also has high thermal and electrical conductivity. Physical and chemical properties of aluminum are given in Table 3.3.

Table 3.3 Physical and Chemical Properties of Aluminum [2]

Property	Value
Atomic Weight	26,9815386 gr/mol
Melting Point	660,32^{o}C
Boling point	2467 ^{o}C
Density	2,71 g/cm^3
Electrical resistivity	(20 °C) 28.2 nΩ·m
Thermal conductivity	237 W/(m.K)
Heat of vaporization	294,0 kj/mol
Heat capacity	(25 ^{o}C) 24,2 J/(mol.K)
First Ionization Energy	577.5 kj/mol
Atomic Radius	125pm (0.125nm)

3.3.2 Usage of the Borax with Aluminum

Aluminum metal and its alloys are widely used as an engineering material in industry due to their low density, high specific strength (strength/weight), high corrosion resistance and so on. The mechanical properties of Al metal can be greatly enhanced by incorporating with some chemical elements and forming into an alloy. On the other hand boron ore and its constituents are also widely used in many areas commercially. Boron can be used in different application areas which seem to be different, according to the composition and boron amount. And also boron has a strategic importance for Turkey. Turkey holds the largest boron deposit in the world. Turkey's share of total world boron reserves is 72% as reserves based on B_2O_3.

Boron and aluminum was selected as raw materials to produce Al-B alloy due to their wide application areas, aluminum's superior mechanical and physical properties; easily machined and cast and boron's strategic importance for Turkey.

CHAPTER 4

EXPERIMENTAL PROCEDURE AND SETUP

4.1 INTRODUCTION

In this chapter experimental works carried out at several steps are presented. Materials used in the experiments and applied mechanical tests and metallographic studies on the samples were explained briefly.

4.2 MATERIALS

Materials used in the current study are 99.70% pure commercial aluminum and borax powder. Primary unalloyed aluminum products were obtained from Seydisehir Aluminum (Turkey) and borax was provided from Eti Mine Works (Turkey). They are standardized as ETIAL-7 according to producer and equivalent international Standard is AA 1070 (USA) which has main impurities of iron and silicon (Table 4.1).In this study borax was used as a boron source to produce Aluminum-Boron alloy.

Table 4.1 Chemical Specification of AA 1070 [33]

Component Elements	(% Weight)
Aluminum, Al	99.7
Copper, Cu	0.040
Iron, Fe	0.25
Magnesium, Mg	0.030
Manganese, Mn	0.030
Other, each	0.030
Silicon, Si	0.20
Titanium, Ti	0.030
Vanadium, V	0.050
Zinc, Zn	0.040

Borax, also known as sodium borate, sodium tetraborate, or disodium tetraborate, is an important boron compound, a mineral and a salt of boric acid. It is usually a white powder consisting of soft colorless crystals that dissolve easily in water. Molecular formula of borax is $Na_2B_4O_7 \bullet nH_2O$ (Table 4.2). Borax has a density of 1.73 g/cm^3, a melting point of 743°C (anhydrous borax) and boiling point of 1575°C [36].

Table 4.2 Chemical Specification of Borax Powder [26]

Component	**Unit**	**Content**
B_2O_3	%	36.47 min.
Purity	%	99.90 min.
Na_2O	%	16.24 min.
SO_4	ppm	70 max.
Cl	ppm	50 max.
Fe	ppm	15 max.

Borax occurs naturally as tincal. Borax can also be produced synthetically from other boron compounds. Naturally occurring borax is refined by a process of re-crystallization [25].

The term borax is often used for a number of closely related minerals or chemical compounds that differ in their crystal water content:

- Anhydrous borax ($Na_2B_4O_7$)
- Borax pentahydrate ($Na_2B_4O_7 \bullet 5H_2O$)
- Borax decahydrate ($Na_2B_4O_7 \bullet 10\ H_2O$)

There are different boron compounds which can be used in this study such as boron oxide and boric acid...etc. But borax is considered as one of the rich boron supplier among the other boron compounds according to literature review. Due to this reason borax was used to supply boron in the experiments.

Pure aluminum and borax were used without any purification and modification in our experiments.

4.3 EXPERIMENTAL METHOD

In this study, conventional casting method was used to produce aluminum-boron alloys and some fabrication parameters were established to compare the mechanical properties of alloys.

The preparation steps of Al-B alloys were considered to consist of three distinct and consecutive steps:

- Aluminum particles and borax powder were placed into the graphite crucible (Figure 4.1) at certain amounts;
- Melting and holding the molten alloy to establish a reaction between the aluminum and borax;
- Casting the molten alloy after the dross was skimmed off from the surface and the melt was stirred thoroughly.

Figure 4.1 Graphite Crucible

A series of experimental trials were carried out in the laboratory in order to compare the effects of prespecified parameters on the mechanical properties of alloys. All these parameters and mutual dependence are demonstrated in Figure 4.2.

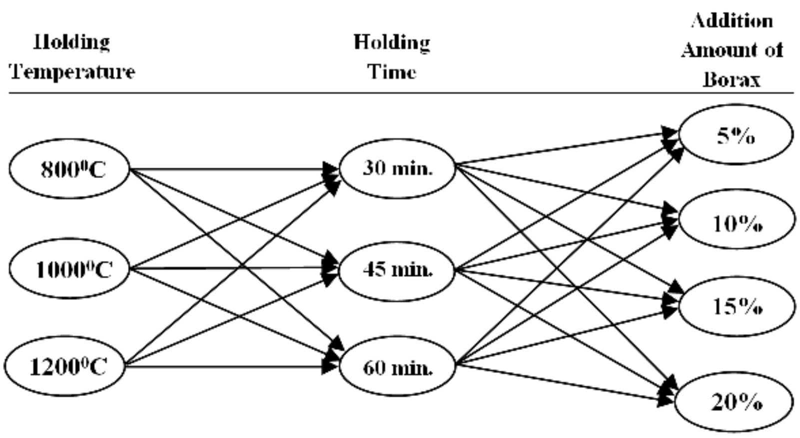

Figure 4.2 Parameters Used in the Experiments

One of the parameter was the additive amount of borax. Borax was used at different amounts of 5, 10, 15 and 20 weight percentages of aluminum. The other parameter was the holding temperature. It was determined as 800, 1000 and 1200^0C to indicate the effect of temperature on the mechanical properties of alloys. The lower limit of 800^0C was a practical limit to maintain the molten alloy as a castable liquid while 1200^0C was identified as the upper limit because of the electrical resistance furnace capacity. The next parameter was holding time. Holding times were established as a 30, 45 and 60 minutes to see the holding time effect on the mechanical properties. According to literature [5] and our trials it is noticed that minimum holding time is at least 30 minutes to complete the reaction between the molten aluminum and viscous glassy borax.

Fabrications of alloys were carried out on an about 400 gram batch scale. AA 1070 aluminum alloy ingots were placed into a graphite crucible with borax powder together and were brought to predetermined temperature using an electrical resistance furnace (Figure 4.3).

Figure 4.3 Electrical Resistance Furnace

The molten alloy thus obtained was held at different temperatures ranging from 800 to 1200^0C for 30 to 60 min. During holding dross was generated on the surface and this dross provided an effective mechanism for protecting the molten alloy from the environmental effects. There was no stirring during holding to avoid excessive oxidation. Before casting into a permanent mold, dross was skimmed off from the surface and the melt was stirred thoroughly with graphite rods for 30 seconds to ensure homogeneity.

Finally, the melt was poured into a metal mold, made by cast iron, with a diameter of 20 mm and a height of 200 mm (Figure 4.4).

Figure 4.4 Permanent Mold

4.4 MECHANICAL TESTS

Mechanical tests play a significant role in evaluating fundamental properties of engineering materials as well as in developing new materials. After producing alloyed samples by conventional casting methods, tensile test, impact test and hardness tests were applied to the samples to compare the effect of the parameters on their mechanical properties.

4.4.1 Tensile Test

Tensile or tension test is a common and important test that provides a variety of information about the mechanical properties of material being tested. Tensile test is widely used to provide basic design information on the strength of materials and is an acceptance test for the specification of materials. Test specimens are machined from the material to be tested in the desired orientation and according to the standards.

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 machined by lathe with a diameter of 15 mm and gauge length 75 mm (Figure 4.5). Tensile test of the samples were carried out by using Shimadzu AGX universal testing device having a capacity of 30 tones (Figure 4.6).

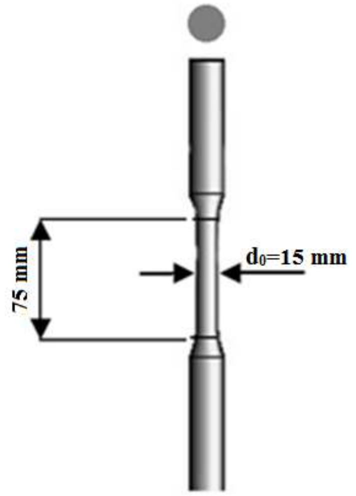

Figure 4.5 Tensile Test Specimen

Figure 4.6 Tensile Test Device

4.4.2 Impact Test

Impact test designed to give information on how a specimen of a known material will respond to a suddenly applied force and determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material's toughness. It is widely applied in industry, since it is easy to prepare and conduct and results can be obtained quickly and cheaply. But a major disadvantage is that all results are only comparative [37]. A notched test specimen is normally employed and the two methods in general use are either the Izod or Charpy test.

In this study, impact test was performed by using Tinius Olsen charpy-type impact test equipment having a capacity of 36.5 kg•m (Figure 4.7). Test specimens were machined according to TS 269 standard with square cross section 10x10 mm and length of 55 mm (Figure 4.8). Notch is an important factor at charpy impact test. The notch in the test specimen affects the results of the impact test directly thus it is necessary for the notch to be of regular dimensions and geometry. All test specimens have “v” type notch with the degree of 45◦ and 2 mm depth.

Figure 4.7 Impact Test Device

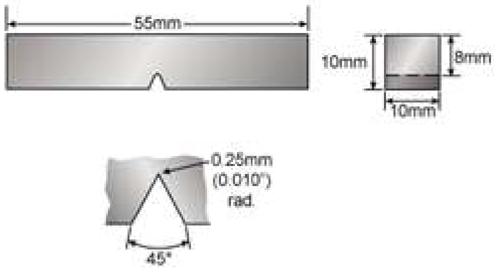

Figure 4.8 Impact test specimen

4.4.3 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 variety of methods used for determining the hardness of materials, but three principal standard test methods are used for expressing the relationship between hardness and the size of the impression, these being Brinell, Vickers, and Rockwell hardness test.

In this study, Hardness tests were performed by using Digirock-RBOV Universal Hardness Tester Device (Figure 4.9). Hardness of the alloys was measured using a Brinell 2.5 mm ball indenter. Test load for each sample was 31.25 kg. Hardness test values were evaluated by averaging the results of 3 successive measurements in all samples.

Figure 4.9 Hardness Test Device

4.5 METALLOGRAPHIC STUDY

Metallography is the science of revealing and evaluating the microstructures of materials. The microstructural characterizations of alloys were involved consecutive steps; sample preparation and microscopic examination.

4.5.1 Sample preparation

Specimen preparation has become a long established art in the fields of metallurgy. Preparation of specimen has 5 steps which are;

- Cutting a sample: must be careful not to significantly alter the microstructure during cutting.
- Mounting: improves handling for small samples.
- Grinding: removes damage introduced by cutting and obtain surface finishes that are ready for polishing.
- Polishing: remove grinding and surface damage.
- Etching: optically enhance microstructural features such as grain size and phase features.

In this work, specimens firstly were cut transversally in a band saw with a height of 20 mm then fixed into Weber mounting device (Figure 4.10).

Figure 4.10 Mounting Device

In order to obtain a highly reflective surface for microscopic examination that is free from scratches and damages, the specimens were carefully grinded and polished. All samples were gently grinded with 240, 400, 600, 800, 1000, and 2000 mesh SiC emery papers. Afterwards samples were roughly polished by 6μ and 1μ diamond solutions and then final polishing was performed by colloidal silica to obtain highly reflective surface. All grinding and polishing operations were made with Metkon Forcipol 2V grinding and polishing machine (Figure 4.11). Microscopic examination of a properly polished, unetched specimen reveals only a few structural features such as inclusions and cracks or other physical imperfections. Etching is carried on to highlight microstructural features. All samples were chemically etched with 0.5 ml hydrofluoric acid (HF) in 100 ml pure water for 45 seconds.

Figure 4.11 Grinding and Polishing Machine

4.5.2 Microscopic examination

Microstructures of the alloyed samples were investigated by computer equipped Nikon MA-100 metallographic microscope (Figure 4.12). Microscopic images were captured by using a "Kameram" digital camera and software.

Figure 4.12 Optical Microscope

CHAPTER 5

RESULTS AND DISCUSSION

5.1 INTRODUCTION

In this chapter, mechanical properties of boron added aluminum compounds are investigated in detail. As a mechanical test, tension, impact and hardness tests were carried out. In order to clarify the microstructural changes between different specimens, microscopic images are also given in this chapter. Experimental results are discussed according to the parameters such as amount of borax addition, holding temperature and holding time.

5.2 RESULTS

Mechanical properties of a material are those properties that involve a reaction to an applied load. The most common properties considered are strength, hardness and impact resistance. General range of usefulness of a material are determined according to these properties [37].

In this study, Al-B alloys have been successfully produced with conventional casting method using some parameters such as, the amount of borax addition, holding temperature and holding time. After producing samples, they are subjected to tensile, impact and hardness tests. Metallographic examinations were also carried out to investigate the variations in the microstructure. Mechanical test results were then compared to evaluate the effect of the parameters on mechanical properties of the Al-B alloys. Test results of samples with and without incorporating borax are presented in Table 5.1.

Table 5.1 Experimental Results

Sample No	Additive	Borax Addition (%)	Holding Temperature (^0C)	Holding Time (min)	Tensile Strength (MPa)	Impact (kg.m)	Hardness (HB) (kgf/mm^2)
1	Pure Aluminum		800	30	52,2	4,7	16,7
2				45	58,7	5,0	17,7
3				60	55,9	4,5	17,2
4			1000	30	54,7	4,9	16,4
5				45	59,7	5,7	17,4
6				60	53,1	4,1	16,9
7			1200	30	49,1	1,1	15,9
38				45	51,1	1,2	16,2
9				60	50,9	0,9	15,4
10	Borax	%5	800	30	62,0	6,0	22,2
11				45	63,6	6,6	23,4
12				60	67,0	6,2	23,0
13			1000	30	64,6	6,2	25,9
14				45	**79,5**	**6,9**	22,6
15				60	73,5	6,5	24,2
16			1200	30	59,7	1,8	22,8
17				45	72,6	2,1	23,4
18				60	70,5	3,3	26,9
19		%10	800	30	62,7	5,9	25,5
20				45	71,8	6,2	**27,4**
21				60	67,7	5,5	22,6
22			1000	30	72,8	6,6	23,0
23				45	78,1	6,8	25,5
24				60	75,3	6,7	26,4
25			1200	30	61,1	1,1	20,8
26				45	73,7	1,2	25,0
27				60	63,7	1,1	19,8
28		%15	800	30	66,8	6,2	24,2
29				45	73,0	6,7	25,0
30				60	75,1	6,6	24,6
31			1000	30	73,8	6,1	23,6
32				45	76,0	6,7	23,8
33				60	75,2	6,0	24,2
34			1200	30	50,5	1,3	22,6
35				45	53,2	1,0	21,8
36				60	58,8	0,9	19,6
37		%20	800	30	71,2	6,4	24,6
38				45	70,6	6,4	25,5
39				60	67,7	6,5	25,0
40			1000	30	66,0	6,1	21,8
41				45	68,5	5,7	26,4
42				60	63,5	5,4	25,0
43			1200	30	52,3	1,2	20,8
44				45	56,4	1,4	22,6
45				60	54,7	1,4	21,8

5.2.1 Tensile Test Results

Tensile behaviors of fabricated Al-B alloys were examined by conducting tensile tests on the specimens. Tensile properties in terms of amount of borax addition, holding temperature and holding time were put into perspective. All of these effecting parameters are presented in the following sections.

5.2.1.1 Effect of Borax Addition

Different amounts of borax were incorporated with aluminum. Borax addition was performed within 5, 10, 15 and 20 weight percentage of pure aluminum. Table 5.2 is the just summary of Table 5.1 in accordance with the borax addition.

Table 5.2 Tensile Test Results According to Borax Addition

	Pure			%5 Borax			%10 borax			%15 Borax			%20 borax		
	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)
30 min	52,2	54,7	49,1	62,0	64,6	59,7	62,7	72,8	61,1	66,8	73,8	50,5	71,2	66	52,3
45 min	58,7	59,7	51,1	63,6	79,5	72,6	71,8	78,1	73,7	73,0	76,0	53,2	70,6	68,5	56,4
60 min	55,9	53,1	50,9	67,0	73,5	70,5	67,7	75,3	63,7	75,1	75,2	58,8	67,7	63,5	54,7

The data reveal that certain amount of borax addition increases ultimate tensile strength of pure aluminum. Figure 5.1 represents the variations of ultimate tensile strength of the samples according to borax addition with respect to holding temperature and time.

All specimens obtained from 800^{0}C and 30 minutes have comparatively higher UTS values with the increase in borax amount. However, when the holding time is increased to 45 and 60 minutes, it is seen that 5, 10 and 15 weight percentage of borax addition increase UTS value while 20% decreases.

At 1000^{0}C, addition of borax generally improves the UTS values. But, this improvement shows different characteristics. For example, at 30 minutes it is observed that UTS values increase continuously from 5% to 15% borax addition but falls at 20%. On the other hand, for 45 and 60 minutes UTS values increase with 5% and almost remain constant up to 15%. Addition of 20% borax decreases UTS for all cases.

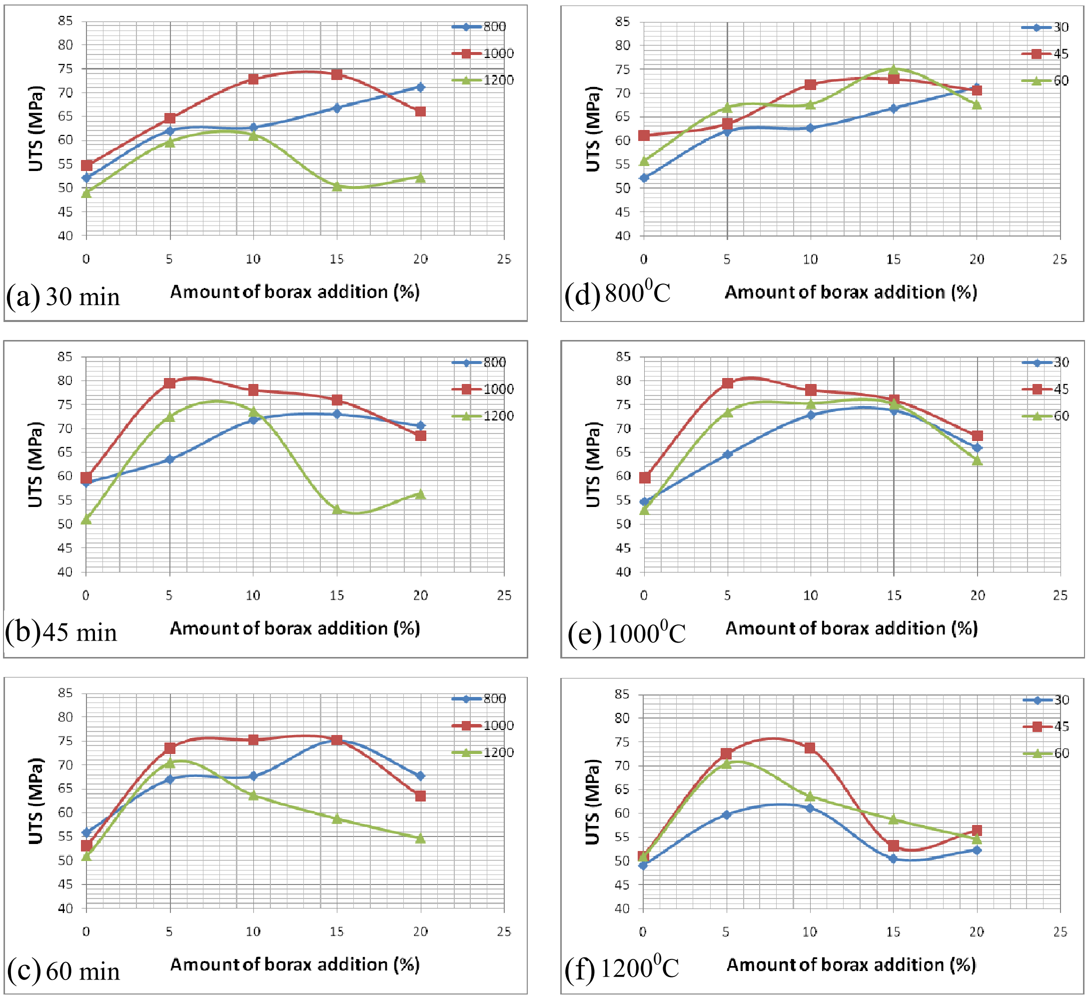

Figure 5.1 Effect of Borax Addition on UTS;
According to a) 30 minutes b) 45 minutes c) 60 minutes
d) 800^0C e) 1000^0C f) 1200^0C.

At 1200^0C up to 10% of borax addition increase in UTS was observed in both 30 and 45 minutes. But after 10 percent of borax addition, UTS values decrease within considerable amount. On the other hand, as far as 60 minute holding time is concerned for 1200^0C, only 5 percent of borax addition increases the tensile strength from 50.9 MPa to 70.5 MPa. One important point was drawn from the results is that, increment of borax addition more than 5 percent has no augmented UTS value.

5.2.1.2 Effect of Holding Temperature

In this section, the effect of holding temperature on the tensile strength of alloyed samples is examined. Three temperatures, 800, 1000 and 1200^0C were carried out during casting process. Table 5.3 indicates all UTS results according to holding temperature with respect to borax addition and holding time

Table 5.3 Tensile Test Results According to Holding Temperature

	800^0C			1000^0C			1200^0C		
	30 min	45 min	60 min	30 min	45 min	60 min	30 min	45 min	60 min
Pure	52,2	58,7	55,9	54,7	59,7	53,1	49,1	51,1	50,9
5%	62,0	63,6	67	64,6	79,5	73,5	59,7	72,6	70,5
10%	62,7	71,8	67,7	72,8	78,1	75,3	61,1	73,7	63,7
15%	66,8	73,0	75,1	73,8	76,0	75,2	50,5	53,2	58,8
20%	71,2	70,6	67,7	66,0	68,5	63,5	52,3	56,4	54,7

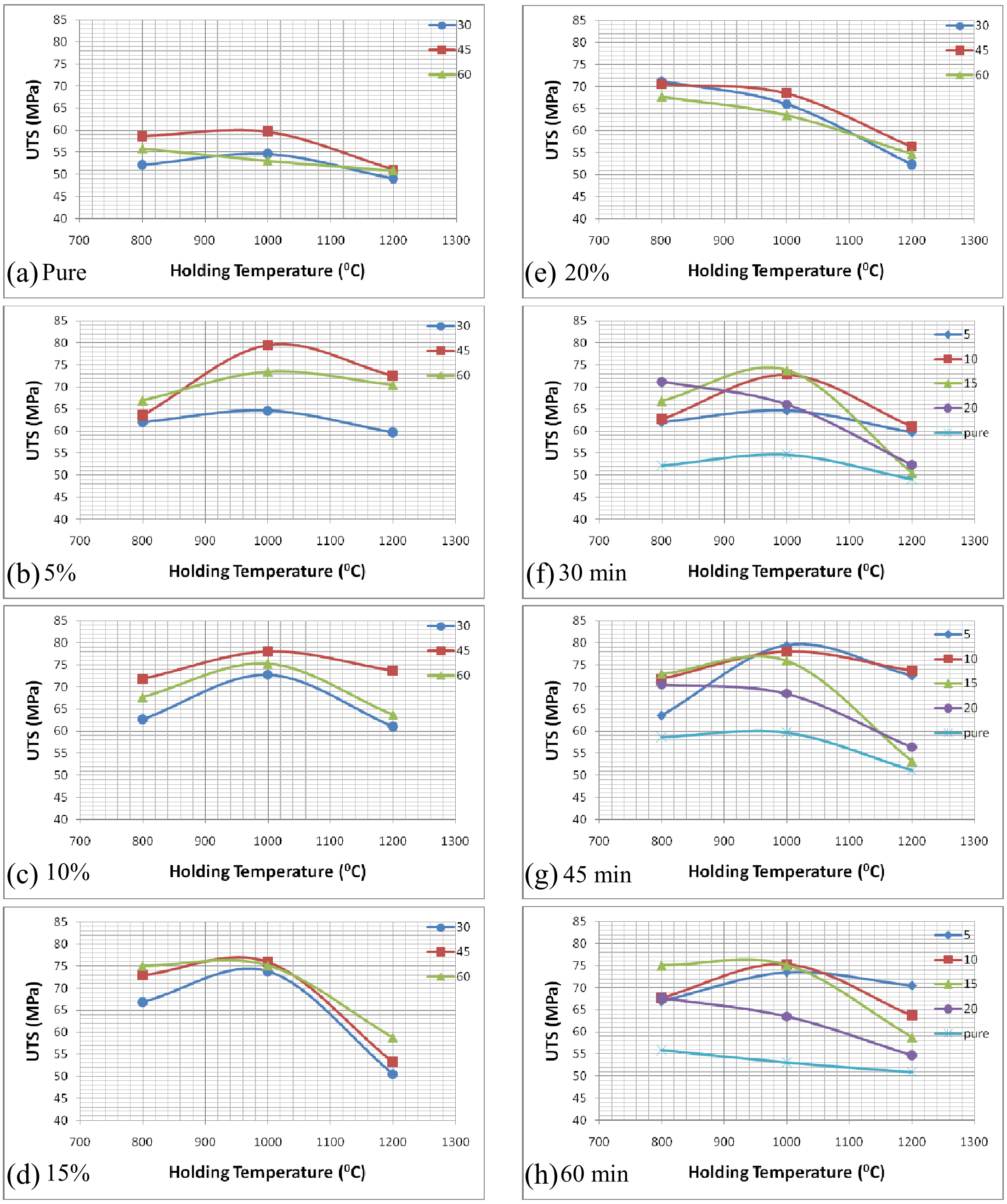

Figure 5.2 Effect of Holding Temperature on UTS; According to
a) pure aluminum b) 5% borax c) 10% borax d) 15% borax e) 20% borax
f) 30 minutes. g) 45 minutes h) 60 minutes

As shown in Figure 5.2, it is very clear that increasing holding temperature from 800^0C to 1000^0C resulted in greater UTS values in all holding times and borax addition except for 20% borax addition. The other clear point is, all UTS values getting worse values for 1200^0C in all conditions.

From Figures 5.2 (f, g and h) it is seen that better UTS values were obtained at higher borax percentages with lower temperatures or lower borax percentages with higher temperatures.

In other words, in order to get higher UTS values, borax should be added to aluminum more than 15% for 800^0C and less than 10% for 1200^0C.

5.2.1.3 Effect of Holding Time

Additional experiments were performed to identify the effect of holding time on the tensile strength of alloys. Three holding times, 30, 45 and 60 minutes, were applied during experiments. The ultimate tensile strength variations according to holding time with respect to borax addition and holding temperature are listed in Table 5.4.

Table 5.4 Tensile Test Results According to Holding Time

	30 minutes					45 minutes					60 minutes				
	Pure	5%	10%	15%	20%	Pure	5%	10%	15%	20%	Pure	5%	10%	15%	20%
800 ^{0}C	52,2	62,0	62,7	66,8	71,2	58,7	63,6	71,8	73,0	70,6	55,9	67,0	67,7	75,1	67,7
1000 ^{0}C	54,7	64,6	72,8	73,8	66,0	59,7	79,5	78,1	76,0	68,5	53,1	73,5	75,3	75,2	63,5
1200 ^{0}C	49,1	59,7	61,1	50,5	52,3	51,1	72,6	73,7	53,2	56,4	50,9	70,5	63,7	58,8	54,7

Figure 5.3 displays the variation of ultimate tensile strength of alloyed samples comparatively in reference to holding temperature depending on borax addition and time.

Holding for more than 45 minutes does not seem to offer any improvement in UTS values in conditions of 10 percentage of borax addition for all holding temperatures as seen in Figure 5.3.

Holding time increasing up to 45 minutes improved UTS values in 5 and 20 percentage of borax addition at 1000^{0}C and 1200^{0}C. In addition to this situation at 800^{0}C, increasing holding time continuously resulted in greater UTS values in 5% while fewer UTS values in 20%.

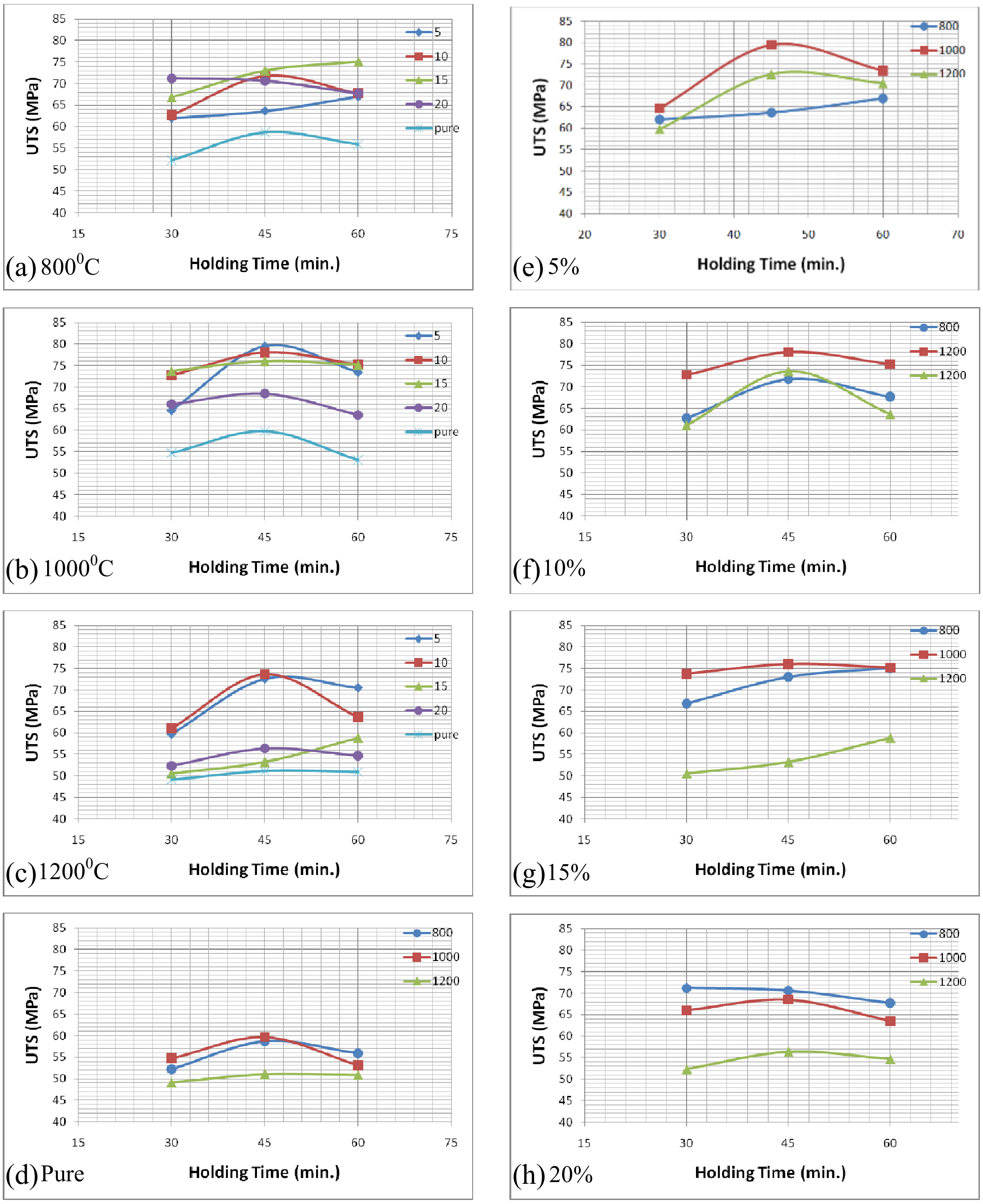

Figure 5.3 Effect of Holding Time on UTS; According to
a) 800^{0}C b) 1000^{0}C c) 1200^{0}C d) Pure aluminum
e) 5% borax f) 10% borax e) 15% borax h) 20% borax

UTS values of the samples incorporated with 15 percentage of borax addition increased continuously when the holding time augmented at 800 and 1200^{0}C. However this increment was observed up to 45 minutes at 1000^{0}C.

5.2.2 Impact Test Results

Charpy impact test were carried out to exam the impact resistance of fabricated Al-B alloys. Impact energy variation of the samples in terms of amount of borax addition, holding temperature and holding time were discussed in the following sections.

5.2.2.1 Effect of Borax Addition

Impact resistance values of the alloyed samples according to borax addition with respect to holding temperature and time are listed in Table 5.5.

Table 5.5 Impact Test Results According to Borax Addition

	Pure			%5 Borax			%10 borax			%15 Borax			%20 borax		
	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)	800 (^{0}C)	1000 (^{0}C)	1200 (^{0}C)
30 min	4,7	4,9	1,1	6,0	6,2	1,8	5,9	6,6	1,1	6,2	6,1	1,3	6,4	6,1	1,2
45 min	5,0	5,7	1,2	6,6	6,9	2,1	6,2	6,8	1,2	6,7	6,7	1,0	6,4	5,7	1,4
60 min	4,5	4,1	0,9	6,2	6,5	3,3	5,5	6,7	1,1	6,6	6	0,9	6,5	5,4	1,4

Data indicate that certain amount of borax addition to the pure aluminum yielded an improvement in impact resistance. The impact energy values are plotted as a function of borax addition for varying holding temperatures and times in Figure 5.4.

Impact energy value of the samples increased slightly with increasing borax addition and maximum value reached at 20 percentage of borax addition in conditions of holding temperature 800^{0}C and holding times 30 minutes. When increasing holding times to 45 and 60 minutes at the same temperature, maximum value was observed at 15%.

The increase in the addition amount of borax up to 10 percentage resulted in higher impact resistance but further addition of borax caused a reduction in impact resistance at 1000^{0}C for 30 and 60 minutes.

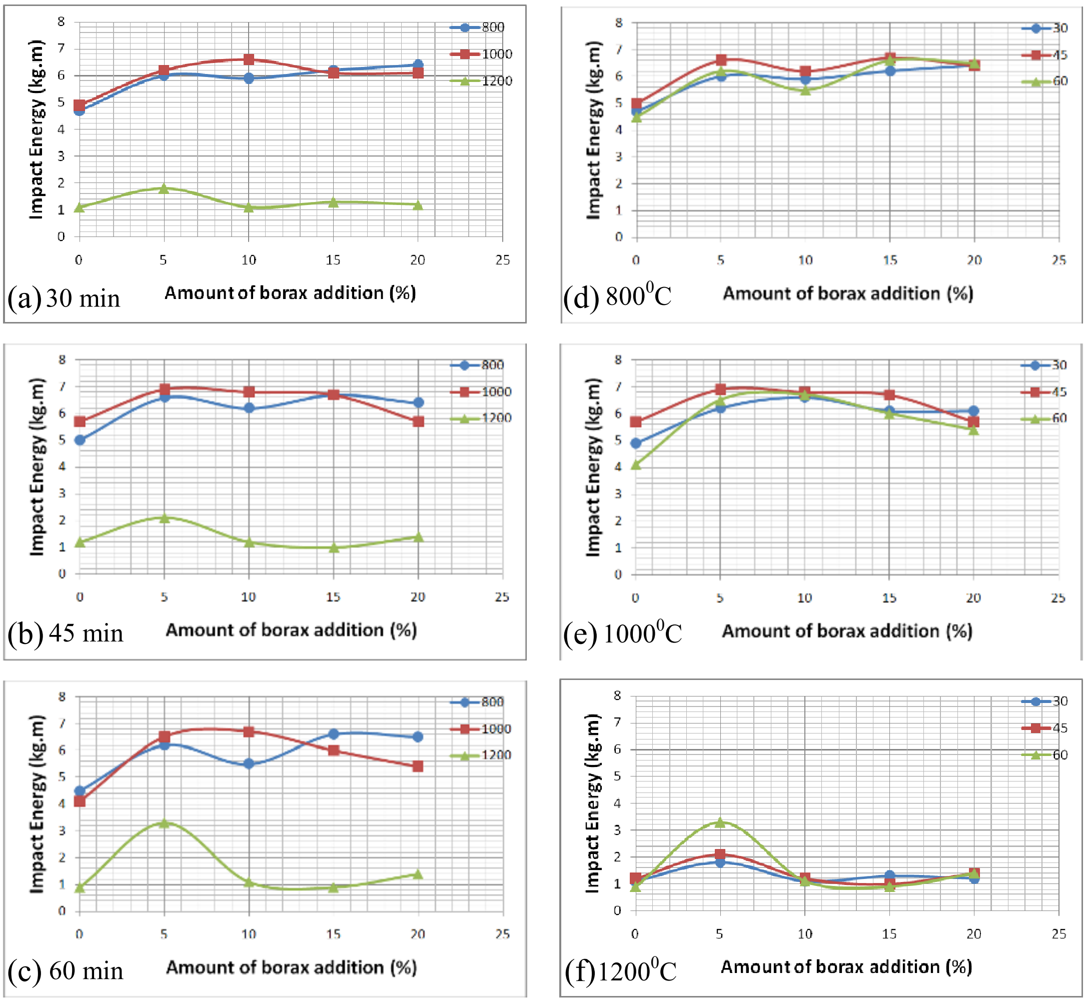

Figure 5.4 Effect of Borax Addition on Impact Energy; According to a) 30 minutes b) 45 minutes c) 60 minutes d) 800^{0}C e) 1000^{0}C f) 1200^{0}C

Impact energy value increased sharply with addition of 5 percentage of borax to the aluminum and at higher borax addition impact energy value kept almost constant except for 20% at 1000^{0}C for 45 minutes.

5.2.2.2 Effect of holding temperature

Impact energy value variations of the fabricated alloys are summarized according to holding temperature in Table 5.6.

Variations of the impact energy value of the samples as a function of holding temperature for varying borax addition and holding times are displayed in Figure 5.5. It indicates that there is strong relationship between the holding temperature and impact energy.

Table 5.6 Impact Test Results According to Holding Temperature

	800⁰C			1000⁰C			1200⁰C		
	30 min	45 min	60 min	30 min	45 min	60 min	30 min	45 min	60 min
0%	4,7	5,0	4,5	4,9	5,7	4,1	1,1	1,2	0,9
5%	6,0	6,6	6,2	6,2	6,9	6,5	1,8	2,1	3,3
10%	5,9	6,2	5,5	6,6	6,8	6,7	1,1	1,2	1,1
15%	6,2	6,7	6,6	6,1	6,7	6,0	1,3	1,0	0,9
20%	6,4	6,4	6,5	6,1	5,7	5,4	1,2	1,4	1,4

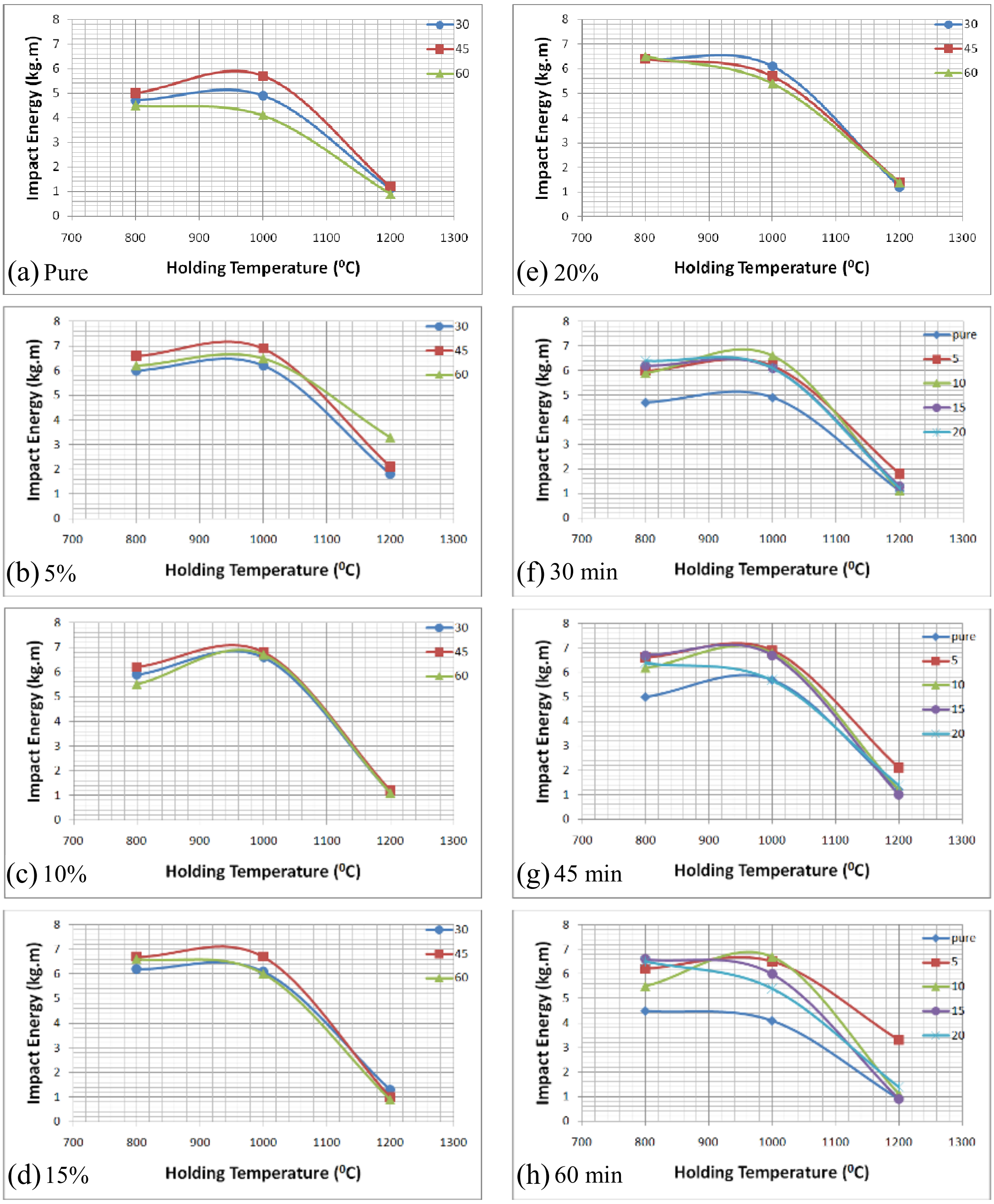

Figure 5.5 Effect of Holding Temperature on Impact Energy; According to a) pure aluminum b) 5% borax c) 10% borax d) 15% borax e) 20% borax f) 30 minutes. g) 45 minutes h) 60 minutes

Increasing holding temperature from 800^0C to 1000^0C generally increased impact resistance except for 15 and 20 percentage of borax addition. At 15%, impact energy value kept almost constant for holding times 30 and 45 minutes while for 60 minutes decreased. Also increase in the holding temperature cause a reduction in impact resistance at 20%.

A significant point was observed from the impact test results, increasing holding temperature to 1200^0C resulted in a dramatic reduction in impact resistance for both pure aluminum and alloyed samples in all conditions.

5.2.2.3 Effect of holding time

Impact test results are arranged in an order according to holding times 30, 45 and 60 minutes in Table 5.7.

Table 5.7 Impact Test Results According to Holding Time

	30 minutes					45 minutes					60 minutes				
	0%	5%	10%	15%	20%	0%	5%	10%	15%	20%	0%	5%	10%	15%	20%
800 ^{0}C	4,7	6,0	5,9	6,2	6,4	5,0	6,6	6,2	6,7	6,4	4,5	6,2	5,5	6,6	6,5
1000 ^{0}C	4,9	6,2	6,6	6,1	6,1	5,7	6,9	6,8	6,7	5,7	4,1	6,5	6,7	6,0	5,4
1200 ^{0}C	1,1	1,8	1,1	1,3	1,2	1,2	2,1	1,2	1,0	1,4	0,9	3,3	1,1	0,9	1,4

Figure 5.6 depicts the variation of the impact energy as a function of holding time for varying borax addition and holding times.

As shown in Figure 5.6, increasing holding time from 30 minutes to 45 minutes resulted in greater impact energy values in all conditions except for 1200^0C and 20 percentage of borax addition. 20% borax addition shows different characteristics at different holding temperatures. For 20% as the holding time was increased, impact energy value almost kept constant at 800^0C while decreased continuously at 1000^0C.

Holding for more than 45 minutes has almost completely negated the impact energy value of the samples in all conditions as seen in Figure 5.6.

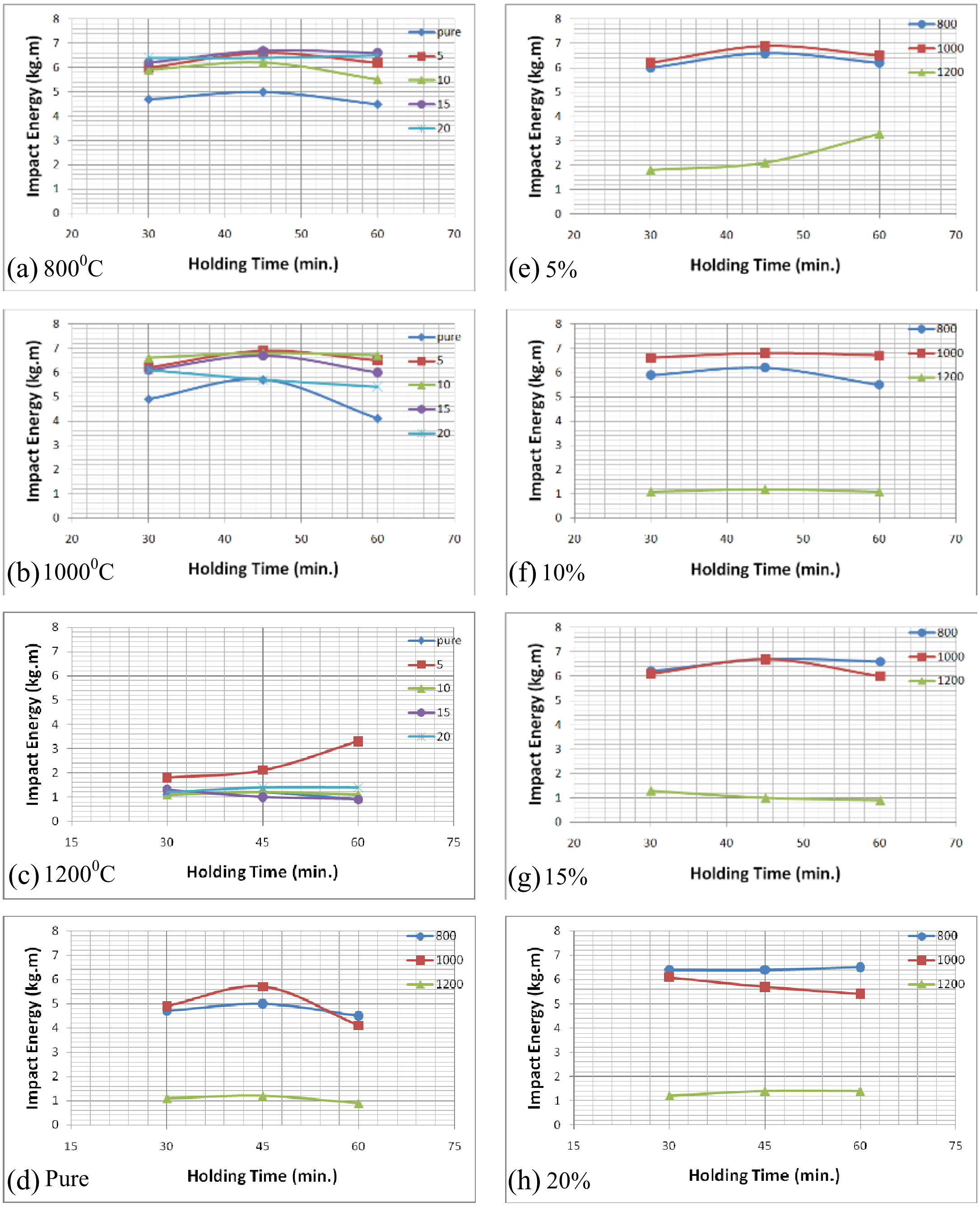

Figure 5.6 Effect of Holding Time on Impact energy; According to a) 800^0C b) 1000^0C c) 1200^0C d) Pure aluminum e) 5% borax f) 10% borax e) 15% borax h) 20% borax

5.2.3 Hardness test results

Brinell hardness tests were conducted on the Al-B alloys to assess the hardness value of the samples. The hardness tests were conducted on the static tests specimens. Applied test load for each sample was 31.25 kg and Brinell 2.5 mm ball indenter was used. Results of hardness tests were evaluated by averaging the results of 3 successive measurements in all samples.

5.2.3.1 Effect of borax addition

Brinell hardness (HB) test results are indicated according to borax addition are listed in Table 5.8.

Table 5.8 Hardness Test Results According to Borax Addition

	Pure			%5 Borax			%10 borax			%15 Borax			%20 borax		
	800 ^{0}C	1000 ^{0}C	1200 ^{0}C	800 ^{0}C	1000 ^{0}C	1200 ^{0}C	800 ^{0}C	1000 ^{0}C	1200 ^{0}C	800 ^{0}C	1000 ^{0}C	1200 ^{0}C	800 ^{0}C	1000 ^{0}C	1200 ^{0}C
30 min	16,7	16,4	15,9	22,2	25,9	22,8	25,5	23	20,8	24,2	23,6	22,6	24,6	21,8	20,8
45 min	17,7	17,4	16,2	23,4	22,6	23,4	27,4	25,5	25	25	23,8	21,8	25,5	26,4	22,6
60 min	16,9	17,2	15,4	23	24,2	26,9	22,6	26,4	19,8	24,6	24,2	19,6	25	25	21,8

Hardness test results of these alloyed samples are in all case better than the pure aluminum ones. The variations in the measured hardness values as a function of borax addition for varying holding temperature and time are respectively plotted in Figures 5.7.

Increased borax addition at 800^{0}C resulted in higher HB value for 30 and 45 minutes. However, this increase is continued till %10 borax addition for both. On the other hand increase in the HB value was only observed between pure aluminum and %5 at 1000 and 1200^{0}C for 30 minutes.

HB value of the samples increased slightly with increasing borax addition and maximum value reached at 20 percentage of borax addition in conditions of holding temperature 800^{0}C and holding times 60 minutes. A similar trend was observed also at 1000^{0}C for 45 minutes.

HB value increased sharply with addition of 5 percentage of borax to the pure aluminum at 1200^{0}C for 60 minutes. However, further addition of borax resulted in a dramatic reduction in HB value at the same condition.

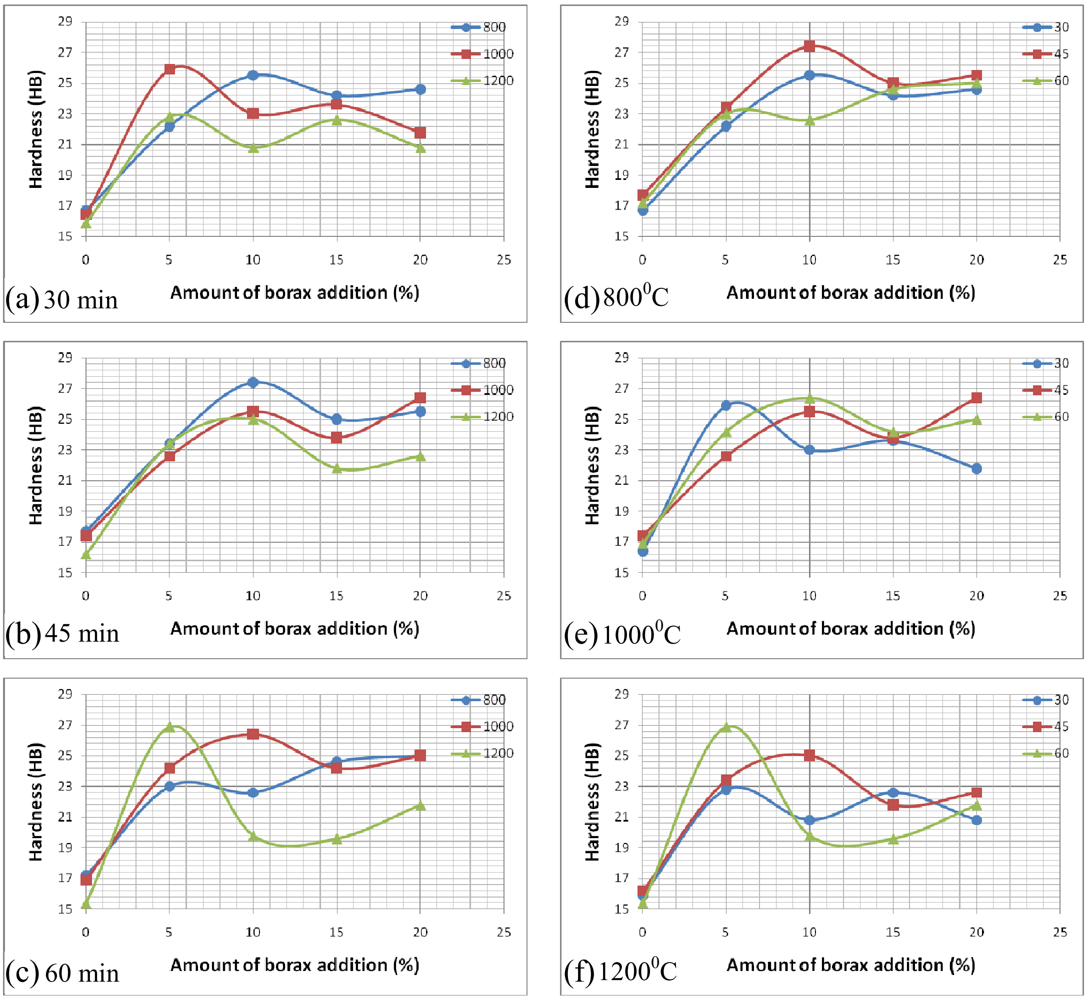

Figure 5.7 Effect of Borax Addition on HB; According to a) 30 minutes b) 45 minutes c) 60 minutes d) 800^{0}C e) 1000^{0}C f) 1200^{0}C.

5.2.3.2 Effect of holding temperature

Brinell hardness value variations of the fabricated alloys are summarized in reference to holding temperature for varying borax addition and holding time in Table 5.9.

Table 5.9 Hardness Test Results According to Holding Temperature

	800^{0}C			1000^{0}C			1200^{0}C		
	30 min	45 min	60 min	30 min	45 min	60 min	30 min	45 min	60 min
0%	16,7	17,7	16,9	16,4	17,4	17,2	15,9	16,2	15,4
5%	22,2	23,4	23,0	25,9	22,6	24,2	22,8	23,4	26,9
10%	25,5	27,4	22,6	23,0	25,5	26,4	20,8	25,0	19,8
15%	24,2	25,0	24,6	23,6	23,8	24,2	22,6	21,8	19,6
20%	24,6	25,5	25,0	21,8	26,4	25,0	20,8	22,6	21,8

Variation of the HB value of the alloys as a function of holding temperature for varying borax addition and holding time are displayed comparatively in Figure 5.8.

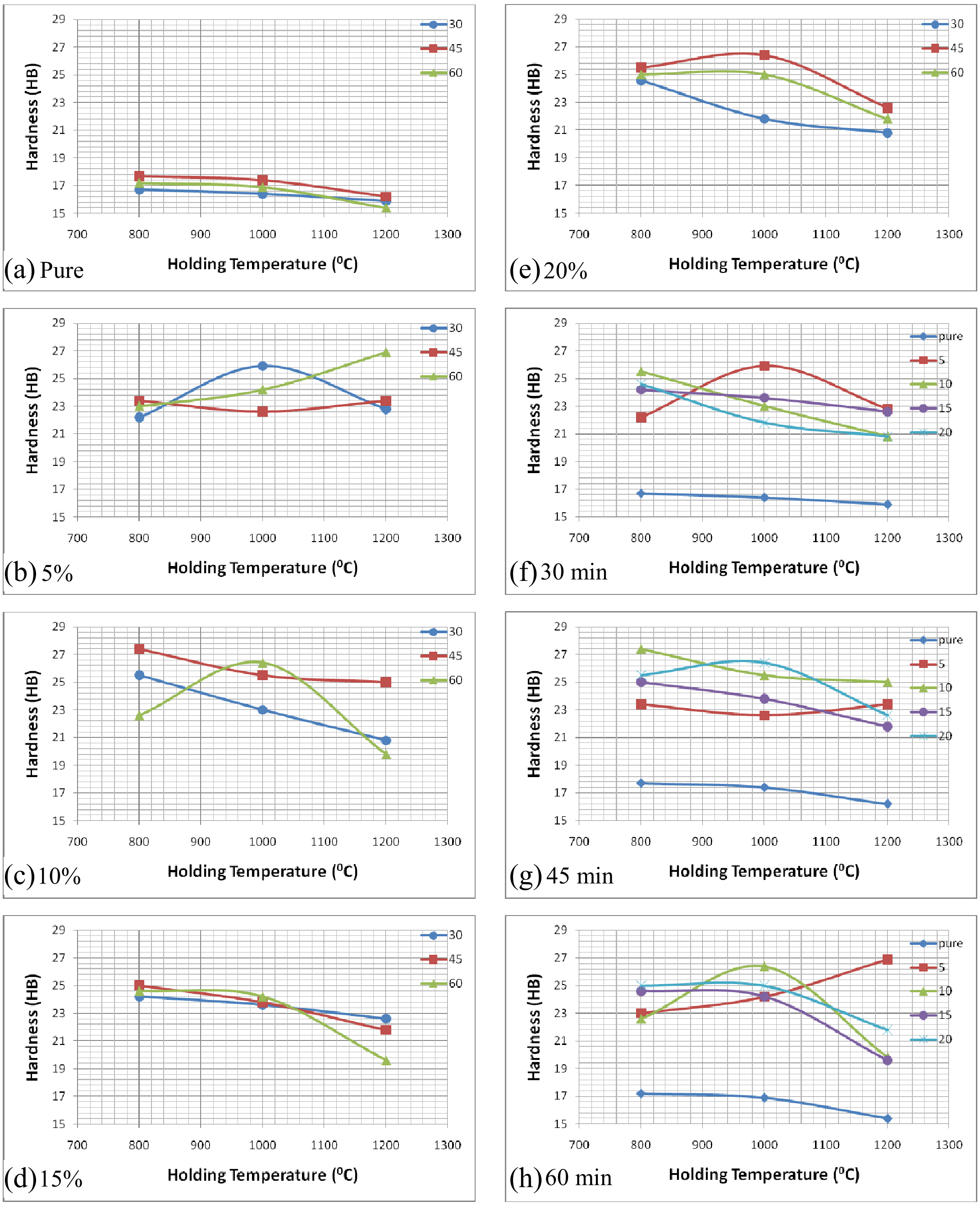

Figure 5.8 Effect of Holding Temperature on HB; According to a) pure aluminum b) 5% borax c) 10% borax d) 15% borax e) 20% borax f) 30 minutes. g) 45 minutes h) 60 minutes

Increasing holding temperature has almost completely negated the HB value of the samples in all conditions except for some conditions as seen in Figure 5.8. Contrary to this, HB value was increased linearly with increasing temperature at 5% borax addition for 60 minutes.

Increasing holding temperature from 800^{0}C to 1000^{0}C increased HB value in conditions of 5% borax addition for 30 minutes, 10% borax addition for 60 minutes and 20% borax addition for 45 minutes. Increase in the holding temperature more than 1000^{0}C cause a reduction in HB value in these conditions.

5.2.3.3 Effect of holding time

Hardness test results are arranged in an order according to holding times 30, 45 and 60 minutes in Table 5.10.

Table 5.10 Hardness Test Results According to Holding Time

	30 minutes					45 minutes					60 minutes				
	0%	5%	10%	15%	20%	0%	5%	10%	15%	20%	0%	5%	10%	15%	20%
800^{0}C	16,7	22,2	25,5	24,2	24,6	17,7	23,4	27,4	25,0	25,5	16,9	23,0	22,6	24,6	25,0
1000^{0}C	16,4	25,9	23,0	23,6	21,8	17,4	22,6	25,5	23,8	26,4	17,2	24,2	26,4	24,2	25,0
1200^{0}C	15,9	22,8	20,8	22,6	20,8	16,2	23,4	25,0	21,8	22,6	15,4	26,9	19,8	19,6	21,8

Holding time-Hardness Brinell value curves are presented in Figure 5.9. HB values of the alloyed samples are compared as a function of holding time for varying borax addition and holding temperature in graphics.

All specimens obtained from 800^{0}C have comparatively rise in HB value with the increase in holding time up to 45 minutes. However, increasing holding time from 45 to 60 minutes resulted in decreasing in HB value. Similar trend was observed in pure aluminum and 20% borax addition at both temperatures 1000 and 1200^{0}C.

Increasing holding time improved HB value in 10% of borax addition at 1000^{0}C and in 5% borax addition at 1200^{0}C. Contrary to this increasing holding time resulted in fewer HB values in 5% borax addition at 1000^{0}C and 15% borax addition at 1200^{0}C.

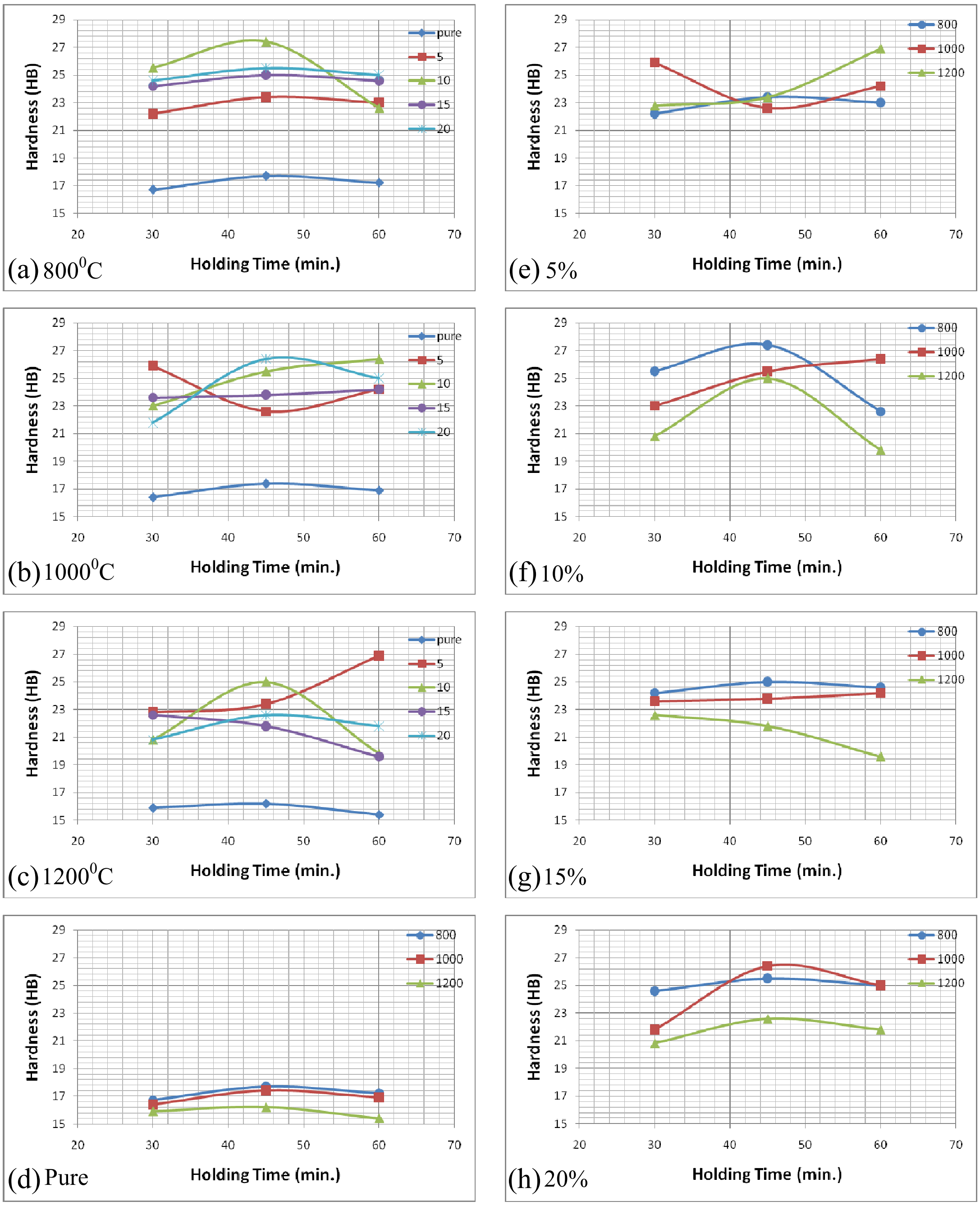

Figure 5.9 Effect of Holding Time on HB; According to
a) 800^{0}C b) 1000^{0}C c) 1200^{0}C d) Pure aluminum
e) 5% borax f) 10% borax e) 15% borax h) 20% borax

5.2.4 Metallographic Study

Optical microscopy observations have been carried out on the Al-B alloys. Polished and etched Al-B samples photographed to investigate their microstructures. Optical photographs are taken in 10x, 50X and 100X scales from optic microscope equipped with camera.

Micrographs show the effects of borax addition to pure aluminum on the microstructure of the cast samples. The formation of aluminum-borides is evidenced by the metallographic study of samples.

The bright region represents the aluminum, the dark gray particles as aluminum-borides in all micrographs.

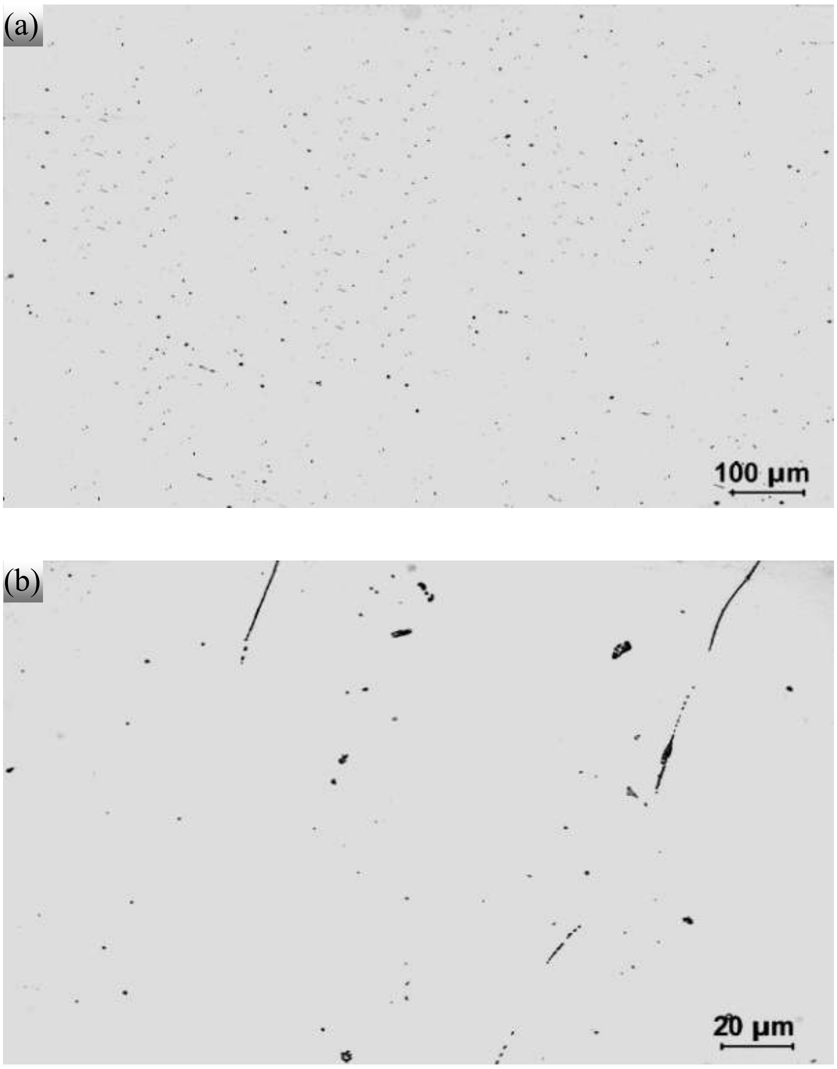

Figure 5.10 Microstructure of Sample 5:
a) 10X b) 50X

Figure 5.10 reveal the microstructure of pure aluminum fabricated at 1000^0C for 45 minutes.

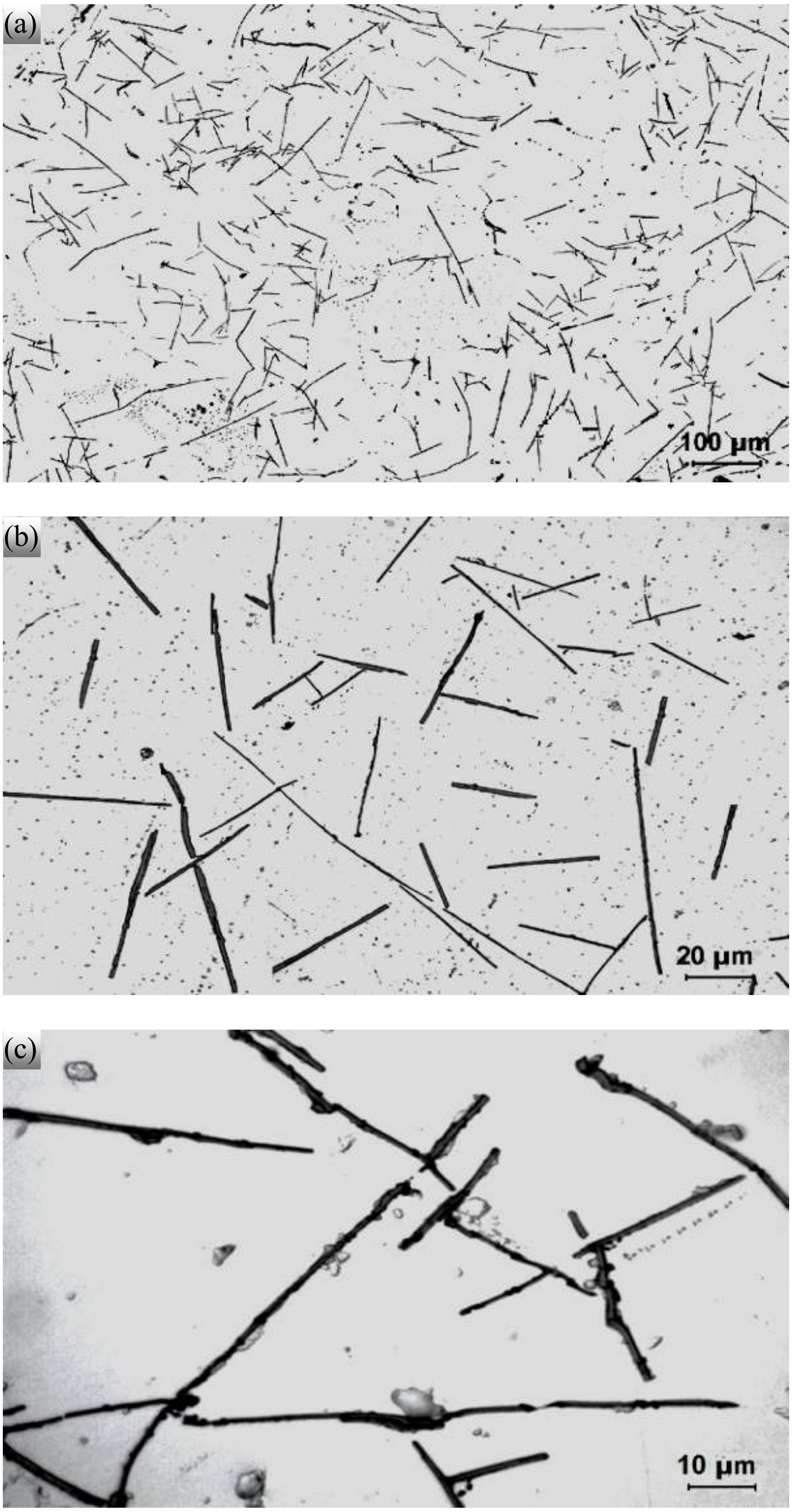

Figure 5.11 Microstructure of Sample 14:
a) 10X b) 50X c) 100X

Figure 5.11 indicate the microstructure of Al-B alloy fabricated with addition of 5 percentage borax addition at 1000^0C for 45 minutes.

A substantial quantity of thicker, longer and larger aluminum-boride flakes are observed in sample 14.

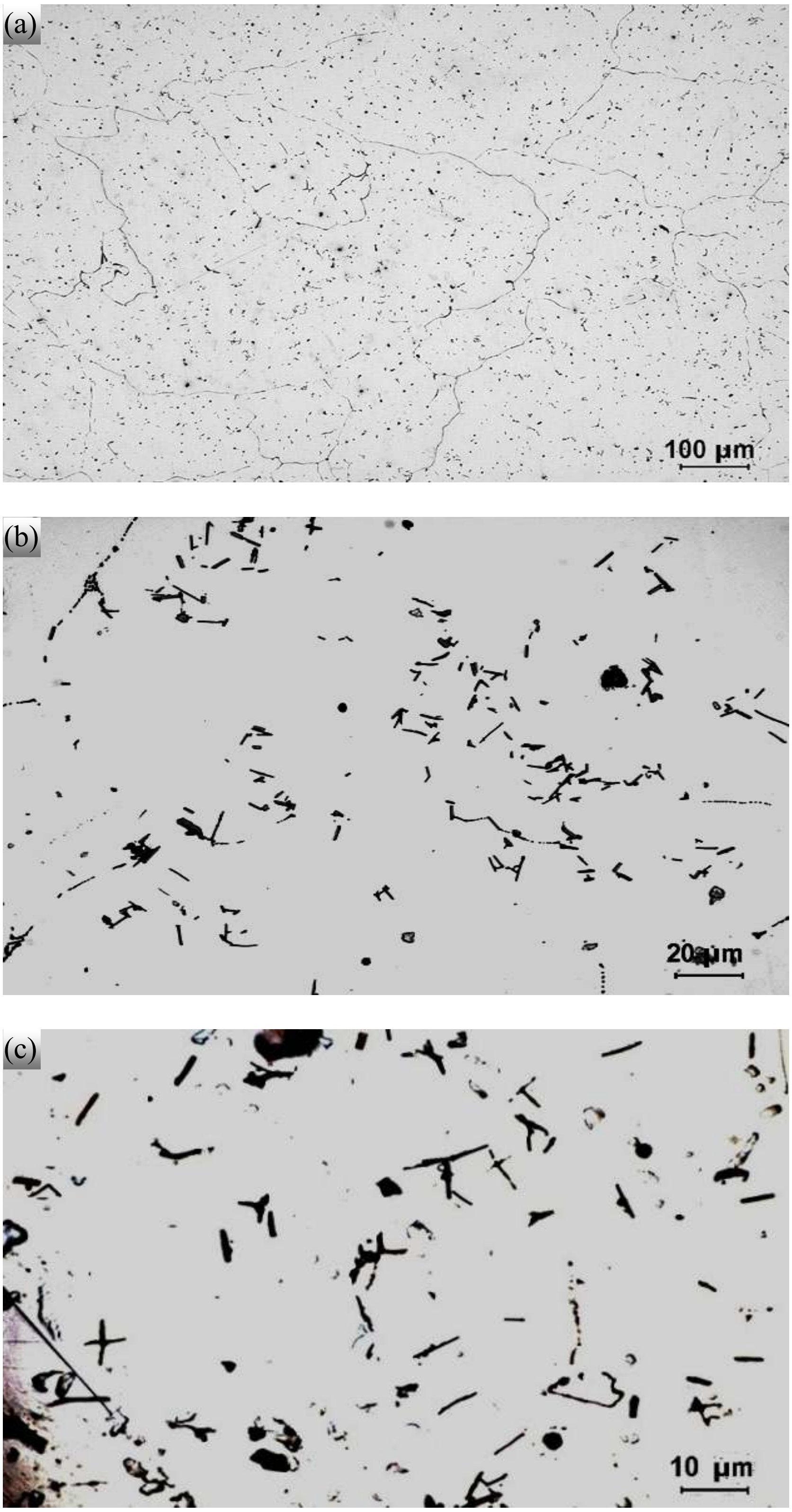

Figure 5.12 Microstructure of Sample 21:
a) 10X b) 50X c) 100X

The sample in Figure 5.12 was prepared by holding 60 minutes at 800^{0}C with the addition of 10% borax. In sample 21, aluminum-boride particles are fewer than sample 14 and have rectangular, cubic and intermediate shape.

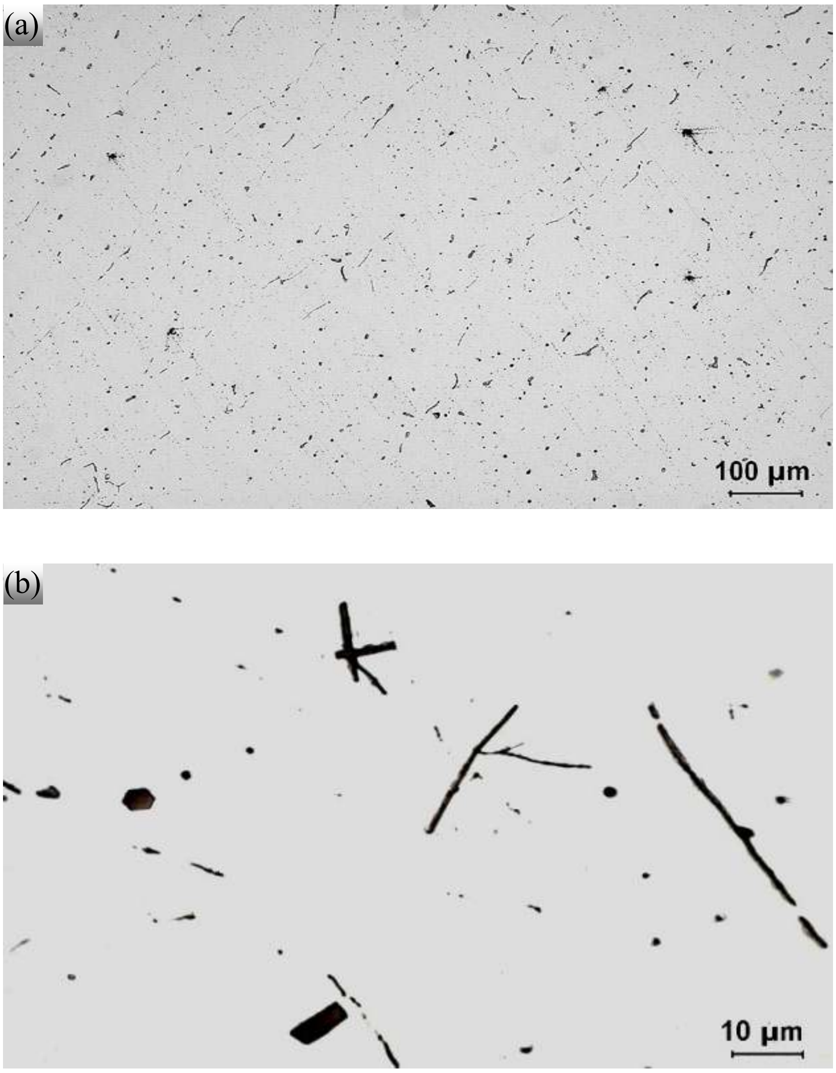

Figure 5.13 Microstructure of Sample 43:
a) 10X b) 100X

Figure 5.13 depicts the microstructure of sample 43, which was produced with addition of 20% percent borax addition at 1200^{0}C for 30 minutes. Hexagonal, rectangular and intermediate shaped aluminum-boride particles were observed in this sample. The population of aluminum-boride particles appeared to be the lower than the others in sample 43.

Microscopic observations of alloyed samples reveal homogeneous distribution of aluminum boride particles in pure aluminum as shown in figures. Microstructural examination of the samples shows that the porosity level of the produced alloys is fewer.

Microstructures of the samples can explain some of the mechanical properties. Sample 14 has much more and larger aluminum-boride flakes than the others and higher UTS, HBN and impact energy value from the others.

5.3 DISCUSSION

Tensile, Hardness and Impact behaviors of pure aluminum follow each other for 45 minutes holding time as seen in tables 5.2, 5.5 and 5.8. In addition, due to change in crystal structure [5], the all the strength behaviors of Al-B alloys are optimum at 1000^0C up to %10 borax addition for 45 minutes.

The temperature 1000^0C is a critical value due to change of phase from AlB_2 to AlB_{12}. So alloys produced at this temperature contains some amount of AlB_2 and AlB_{12} [4]. Flakes of AlB_2 and AlB_{12} caused to higher strength against to various loadings. In fact flakes could not emerge at 800^0C and disappear at 1200^0C (Figure 5.10-12). So 1000^0C is the optimum temperatures for formation of flakes [12].

Aluminum absorbs the borax into crystal structure in different ways. AlB_2 composition has been created as hexagonal structure and AlB_{12} composition has been created as a complex orthorhombic crystal structure [12]. The holding temperature of 1000^0C provided high boron absorption at 45 minutes holding time so these conditions created the best strength characteristics (Figure 5.2).

The highest strength values have been obtained at 5-10% borax addition as seen in Table 5.2, 5.5 and 5.8. The reason is, the excess borax causes more glassy liquid borax and dross during melting operation and they prevent the reaction between aluminum and borax [14].

CHAPTER 6

CONCLUSIONS

The main object of this thesis has been achieved and mechanical properties of Al-B alloys have been described. The achieved results show that the examination of Al-B alloys at various temperatures, holding time and borax ratios is considerable importance.

Those achievements may be summarized as follows:

1. A systematic experimental method has been tested successfully. The management of the casting and the dealing of dross are succeeded.

2. The casted product has been machined according to the tests which will be applied and specimens have been prepared.

3. Various tests have been carried out as tensile, impact and hardness tests due to standards.

4. Sample preparation procedures have been carried out for metallographic investigation.

The obtained and observed results may be concluded as follows:

a. The critical temperature for the flake formation given in the literature has been validated experimentally at 1000^0C. The highest strength values obtained at this temperature.

b. The range of absorption of borax has been described roughly as %5-15. The greatest mechanical test results obtained in this ranges.

c. The holding time for the best mechanical properties has been described at 45 minutes.

d. Tensile, Impact and Hardness behaviors of Al-B alloys follow each other at 1000^0C holding temperature and for 45 minutes holding time.

e. Increase in mechanical properties of the alloyed sample are:
   - 33% for UTS value
   - 21% for impact energy
   - 54% for HB value

   when comparing the maximum values with pure aluminum produced in the same conditions.

RECOMMENDATIONS FOR FUTURE STUDIES

Utilization of the following items will be useful to improve the obtained results of the present work and extend the investigation fields:

- Since casting process was performed under atmospheric condition, alloys can be produced under protected atmosphere.
- In addition to the mechanical properties, grain refinement performance of Al-B alloys can be studied.
- In addition to borax, different boron compounds such as B_2O_3, KBF_4 can be used as a boron source.
- Heat treatment can also be applied on the fabricated alloys.
- Different alloying elements such as, Cu, Ti can be added to improve mechanical properties and increase the boron emission amount in the Al-B alloy.

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