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EFFECT OF CONTROLLED QUENCHING ON THE AGING OF 2024 ALUMINUM ALLOY CONTAINING BORON

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Abstract: *The presence of alloying elements, sometimes in a very small amount, affects mechanical properties; one of these elements is Boron. In Aluminum industries, Boron master alloy is widely used as a grain refiner In this research, the production process of Aluminum –Boron master alloy was studied at first; then, it was concurrently added to 2024 Aluminum alloy. After rolling and homogenizing the resulting alloy, the optimal temperature and time of aging were determined during the precipitation hardening heat treatment by controlled quenching (T6C). Then, in order to find the effect of controlled quenching, different cycles of heat treatment including precipitation heat treatment by controlled quenching (T6C) and conventional quenching (T6) were applied on the alloy at the aging temperature of 110°C. Mechanical properties of the resulting alloy were evaluated after aging at optimum temperature of 110°C by performing mechanical tests including hardness and tensile tests. The results of hardness test showed that applying the controlled quenching instead of conventional quenching in precipitation heat treatment caused reduction in the time of reaching the maximum hardness and also increase in hardness rate due to the generated thermo-elastic stresses rather than hydrostatic stresses and increased atomic diffusion coefficient as well. Tensile test results demonstrated that, due to the presence of boride particles in the microstructure of the present alloy, the ultimate tensile strength in the specimens containing Boron additive increased by 3.40% in comparison with the specimens without such an additive and elongation (percentage of relative length increase) which approximately increased by 38.80% due to the role of Boron in the increase of alloy ductility.*

Keywords: *Aluminum–Boron master alloy, 2024 Aluminum alloy, Precipitation hardening heat treatment, Controlled quenching, Thermo-elastic stress, Thermostatic stress.*

1. INTRODUCTION

Aluminum alloys, series 2000, are widely used in manufacturing airplane parts such as hull, wheel and wing skin and also in transportation and automotive industries like truck wheels and suspension components due to their high strength to weight ratio, good ductility, suitable fracture toughness and excellent thermal stability [1,2,3]. One of the most common alloys that is widely used in aerospace industries is 2024 Aluminum alloy (Al-4.2Cu-1.5 Mg-0.6Mn (Wt.%)) which was introduced in 1930 [4]. In the precipitation hardening alloys, a hardness cycle takes place as a result of increasing the density of dislocations and dislocation –precipitate interaction. In these alloys, hardness cycle would be useful if the formed precipitations are not easily shearable by dislocations [2]. The mechanical strength of alloys depends on the resistance mechanisms against motion of dislocations such as mechanisms of precipitate strengthening, solute strengthening, grain boundary strengthening and strengthening mechanism by dislocation and adding elements to the alloy's microstructure. Up to now, many physical models have been presented, in which alloy elements play an important role in thermodynamics (stable balance or semi-stable) and transfer kinetics. The relationship between strength and microstructure is described through the interaction of dislocations and these alloy elements available in the microstructure. In heat-treatable Aluminum alloys such as 2024 alloy, strengthening mechanism is the precipitate strengthening (formation of precipitates), through which high strength can be obtained [5]. On the other hand, thermo-mechanical treatments led the precipitates formed in the aging process to be finer and to be distributed more uniformly [6].

During the casting process of Aluminum and its alloys, master alloys of Al-B ، Al-Ti and Al-Ti-

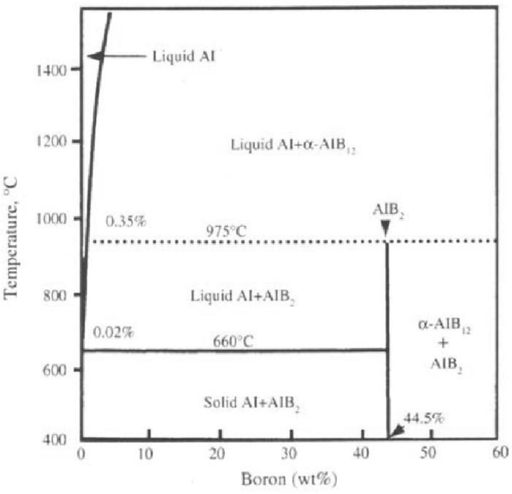

Fig. 1. Aluminum rich end of Aluminum–Boron fuzzy diagram [11]

B are commonly used for fine graining and strength increase [7,8]. Adding Boron Oxide (B_2O_3) to molten Aluminum at 800 ˚C and heating it in the resistance furnace for 30 min is the most common method of producing Al-B master alloy [9]. Binary diagram Al-B (Figure 1) indicates low solubility of Boron element in Aluminum. The solubility line slope in this diagram shows that, when temperature decreases, Boron solid solubility in Aluminum is reduced; therefore, this alloy is hardening perceptible [10].

Referring to the diagram, when the amount of Boron in Aluminum is under 44.50% of weight percent, AlB_2 phase would be stable at room temperature while AlB_{12} phase would be more stable at temperatures above 975˚C [11].

2. EXPERIMENTAL DETAILS

In this research, 2024 Aluminum alloy was used and its chemical compound is mentioned in Table 1.

At first, the above alloy was placed in the resistance melting furnace; then, at 800 ˚C the powder mixture of Aluminum and B_2O_3 wrapped in an Aluminum foil was added to the mentioned molten 2024 Aluminum and stirred by a wooden stirrer. After 10 min, the alloy was cast in a two piece Cast-iron mold with the height of 50mm and the length of 500 mm. In order to determine chemical composition of Boride compounds formed through the alloy making, fuzzy analysis with X ray diffraction (XRD) was conducted on the resulting alloy. The cast alloy was heated at a temperature higher than recrystallization temperature of Aluminum during the hot rolling process and was converted into the sheets with thickness of 2.1 mm through 80% reduction in thickness. The specimens (10*10*2.1 mm) for hardness test were prepared from these sheets. The tensile specimens for tensile test were prepared according to the ASTM E8M standard.

In order to exclude the effect of cold working, the prepared specimens were homogenized for 3 h at 460 ºC. To determine optimal aging temperature for 2024 Aluminum alloy containing Boron, some specimens were solution treated for 1 h at 490 ºC and were exposed to the aging operation immediately after controlled quenching for 1 h at 25 ºC, 50 ºC, 80 ºC, 110 ºC, 150 ºC and 190 ºC; and, in this manner, optimal aging temperature (110 ºC) was specified. The highest hardness of precipitate was achieved at 40 min of aging time [12]. During the hardness test which was performed at aging temperature of 110 ºC, the hardening behavior of the alloy was compared in precipitation hardening heat treatment by both controlled quenching (T6C) and conventional quenching (T6). Then, tensile specimens were solution treated for 1 h at 490 ºC and were immediately exposed to controlled quenching operations. Tensile test was performed on these specimens at room temperature after aging for 40 min at 110 ºC. It is necessary to note that the tensile test was done under similar

Table 1. Chemical composition of the used 2024 Aluminum alloy

Element	Cu	Mg	Mn	Si	Fe	Cr	Ni	Zn	Ti	Al
Wt.%	3.90	1.35	0.41	0.25	0.30	0.02	0.01	0.06	0.004	Rest

conditions on the specimens prepared by the primary alloy (lacking Boron additive) to be compared with the alloy containing Boron additive. Chemical composition was determined by fuzzy analysis with X ray diffraction (XRD). Hardness of the specimens was measured by Brinell hardness tester under the applied load of 62.50 kg (613.125 N) according to ASTM E8 standard. The optical microscope was used to observe and study granulation configuration of the maximum hardness specimens and SEM- TESCAN VEGA electron microscope equipped with the element analysis accessories with Energy Dispersive Spectrometry (EDS) was used to observe the precipitates. Tensile test was performed at room temperature and at the speed of 1 mm/min by Universal tensile tester made by Schenck Trebel.

3. RESULTS AND DISCUSSION

3. 1. Thermo-Dynamical Study on the Reaction of Boron Oxide with Aluminum and the Alloy Microstructure Resulted from Casting

By adding the mixture of Aluminum and Boron Oxide to the molten 2024 Aluminum alloy at 800 °C, the Aluminum Oxide was more stable than the Boron Oxide according to Ellingham Richardson diaphragm; therefore, Aluminum element can reduce Boron Oxide. The following reactions were conducted [9,13]:

$$2Al+B_2\%_3\rightarrow Al_2\%_3+2[B] \qquad \Delta\%^0_{(Al_2\%_3)}=-446226"\ 131.64\,\mathrm{T}(\frac{J}{mol.^0K}) \tag{1}$$

$$3Al+B_2\%_3\rightarrow AlB_2+Al_2\%_3 \qquad \Delta\%^0_{(AlB_2)}=-511783"\ 126.14\,\mathrm{T}(\frac{J}{mol.^0K}) \tag{2}$$

$$Al+2[B]\rightarrow AlB_2 \qquad \Delta\%^0_{(AlB_2)}=-65557-5.5\,\mathrm{T}(\frac{J}{mol.^0K}) \tag{3}$$

$$Al+12[B]\rightarrow AlB_{12} \qquad \Delta\%^0_{(AlB_2)}=-220000-7.5\,\mathrm{T}(\frac{J}{mol.^0K}) \tag{4}$$

By substituting T=1073 °K in the above relations, change in free energy of these reactions would be respectively as follows [9,13]:

$$\Delta\%_{(1)}{}^0{}_{(Al_2\%_3, 1073^{0K})}=-304976.28(\frac{J}{mol.^0K}) \tag{1}$$

$$\Delta\%_{(2)}{}^0{}_{(AlB_2, 1073^{0K})}=-376434.78(\frac{J}{mol.^0K}) \tag{2}$$

$$\Delta\%_{(3)}{}^0{}_{(AlB_2, 1073^{0K})}=-71458.50(\frac{J}{mol.^0K}) \tag{3}$$

$$\Delta\%_{(4)}{}^0{}_{(AlB_{12}, 1073^{0K})}=-211952.50(\frac{J}{mol.^0K}) \tag{4}$$

Considering the above calculation, free energy changes (ΔG°) in all these reactions were negative; so, they were achievable. But, considering the results of ΔG° at 800 °C, reaction 2 was more achievable. XRD analysis on the alloy showed the presence of AlB_2 particles in the alloy composition (Figure 2). Therefore, according to the binary diagram Al-B (Figure 1), the resulting alloy at 800 °C was in 0.02 weight percent solubility section in the Boron field and the extra Boron was precipitated in the grain boundaries as AlB_2 precipitates.

Scanning Electron Microscopy (SEM) of 2024 Aluminum alloy containing Boron in casting state (Figure 3) shows the presence of precipitates in the

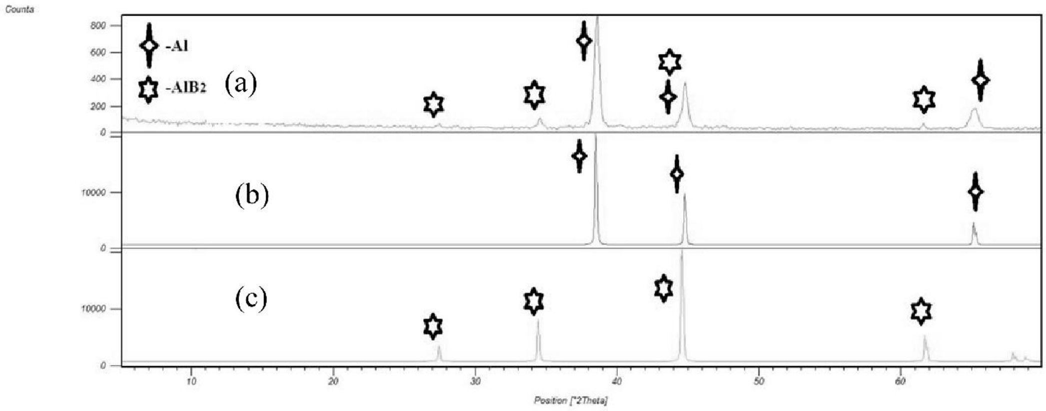

Fig. 2. XRD analysis, a) 2024 Aluminum alloy containing Boron additive resulting from casting after homogenization operations for 3 h at 460 °C, b) pure Aluminum card, c) pure AlB_2 card

grain and intra-granular boundaries in two different magnifications. Figure 4 demonstrates the results of element analysis on these precipitates using Energy Dispersive Spectrometry (EDS). With respect to the binary diagram Al-B (Fig 1) Boron solution is approximately zero at room temperature and at 660 ºC the maximum amount of Boron solution in Aluminum is 0.02%. Consequently with

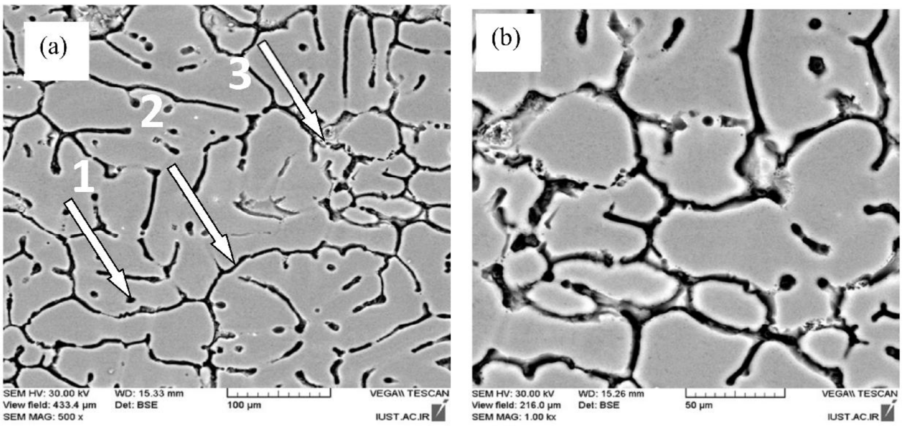

Fig. 3. Scanning Electron Microscopy (SEM) of 2024 Aluminum alloy containing Boron additive in the casting state with hardness of 110 Brinnel, a) 500 times magnification b) 1000 times magnification

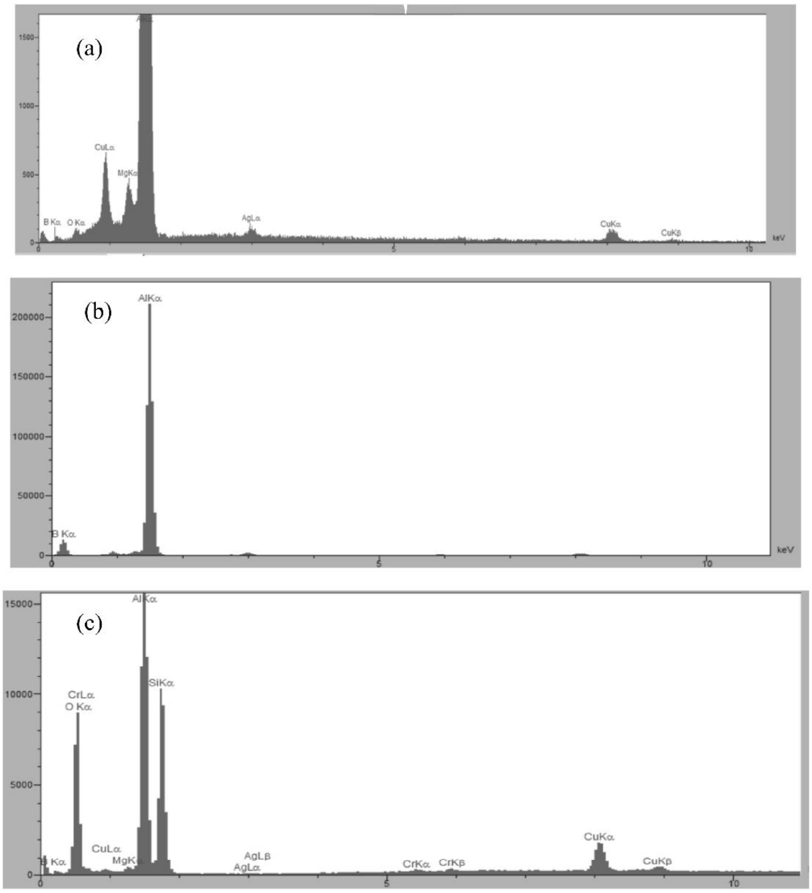

Fig. 4. EDS results of 2024 Aluminum alloy containing Boron additive in casting conditions, a) dark precipitates (arrow 1), b) grain boundary (arrow 2), c) bright precipitates located in the grain boundary (arrow 3), as shown in Figure 4

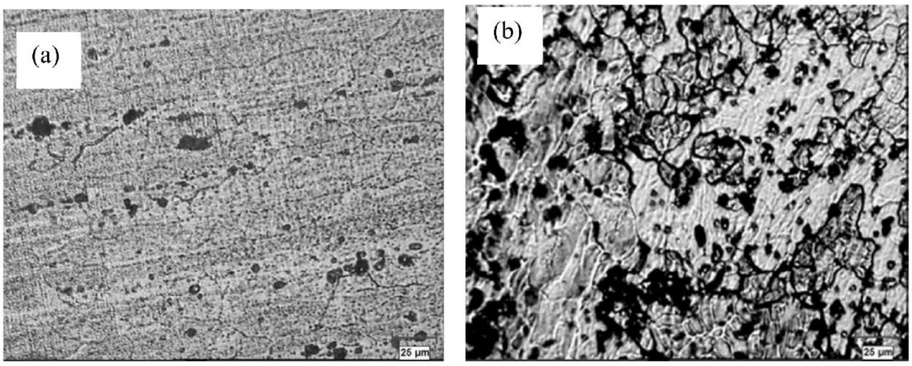

Fig. 5. Optical microscope image of 2024 Aluminum alloy microstructure in casting conditions, a) lacking Boron additive, b) containing Boron additive with 200 times magnification

declining the temperature Boron deposits in the form of rich phase of AlB_2. In relation to the mentioned matter, it is likely that these particles are AlB_2.

Figure 5 compares optical microscope image of 2024 Aluminum alloy microstructure containing Boron additive in the casting state with the primary alloy (lacking Boron additive). Considering that the present inter-metallic particles act as heterogeneous grain refining locations during freezing process and cause relatively fine-grained and coaxial structures, therefore, here micro-boride particles reduced size of the grains by sitting on grain boundaries [7].

3. 2. Determining Optimum Temperature and Aging Time

As shown in Figure 6, the maximum hardness was related to 110 °C with hardness of 134 Brinell; therefore, suitable temperature for aging was considered 110 °C. Due to aging, very fine precipitates were formed, the size and distribution of which were very important. Coarse particles were the reason of weak mechanical properties. Finer precipitates with more volume percent caused higher hardness [14]. On the other hand, considering that the alloy hardening process depends on the penetration and grain refining process, the alloy elements in the

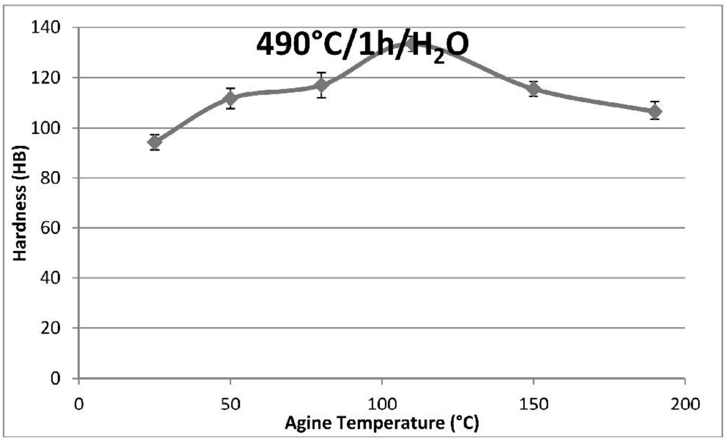

Fig. 6. Hardness diagram in terms of temperature related to the specimens containing Boron solved for 1 hr at 490 °C, controlled quenched in cold water and aged for 1 h at different temperatures (T6C).

alloy play a very effective role in the aging time [15]. So, it seems that the formed precipitates at 110 ºC reached the most suitable distribution and relative volume percent during the aging process and the reason referred to boride compounds.

3. 3. Studying Effect of Controlled Quenching on the Precipitate Hardening of 2024 Aluminum Containing Boron

Figure 7 compares the results of precipitation hardening operations by controlled quenching (T6C) and conventional quenching (T6) at aging temperature of 110 ºC.

As far as the controlled quenching line graph is concerned, the hardness of sample from 73 Brinel (related to super structured solid solution) pretty steeply skyrocketed to the highest point, 136.4 Brinell, only in 40 minutes. By contrast in the line graph related to the conventional quenching, the hardness from 96.7 Brinell (related to super structured solid solution) reached to the maximum hardness with 124.3 Brinell in 90 minutes. The comparison between behaviors of these unfolds that duration of reaching the highest hardness under precipitation hardening operations with controlled quenching (T6C), decreased by about 55.60% in comparison with precipitation hardening operations with conventional quenching (T6);additionally, the hardness level increased by approximately 12%. Behavior of the specimen during the controlled aging in initial stages of aging was noteworthy. Hardness increased from 73 to 112.80 Brinell within the first 5 min that was approximately 54.52% growth.

The reason of reduction in time of reaching the maximum hardness in the controlled quench specimens was the generation of thermo-elastic stresses in the specimens. Conventional quenching, i.e. rapid cooling of the specimen from all sides inside a cooling liquid, created hydrostatic compressive stresses in the part while use of controlled quenching, i.e. rapid cooling of the specimen from a special side, created elastic stresses. The main difference between the

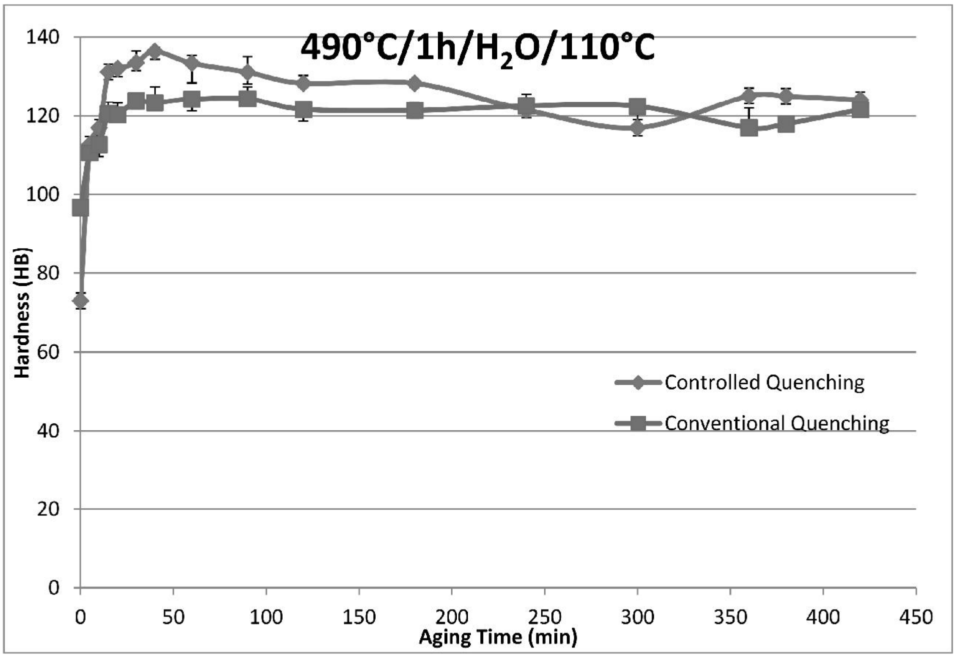

Fig. 7. Hardness diagram in terms of time related to the specimens containing the dissolved Boron for 1 h at 490 ºC, controlled quenched (T6C) and conventional quenched (T6) in cold water during the aging process at aging temperature of 110 ºC

controlled quenching and conventional quenching in the precipitation hardening heat treatment was the generation of thermo-elastic stresses rather than hydrostatic stresses due to temperature gradient. Although conventional quenching created the high-energy regions in the part and suitable supersaturated solid solution for subsequent aging process, the applied hydrostatic stress had a negative effect on diffusion coefficient, grain refining process and growth of precipitates in aging phase. Elastic stresses created elastic energy in the matter. Increase of elastic energy in the matter increased the atomic frequency, orientation of the atomic diffraction and diffusion coefficient. These stresses comprehensively included the specimen and increased the grain fining force of the precipitates in all parts of the specimen .In fact, thermo-elastic stresses increased the energy and tendency to grain fining of the strengthening precipitates in a special direction and, as a result of this, the amount, volume and distribution of the strengthening precipitates in the field were increased compared with the conventional quenching. Thus, the hardness of controlled quenched specimens was raised compared with the conventional quenched ones in a shorter time period [16].

3. 4. Tensile Test

Figure 8 shows results of tensile test in control specimen (without Boron additive and without heat treatment), the specimens without Boron additive and containing Boron additive. As can be observed, the presence of boride compounds in the alloy microstructure increased yield strength by 5.50% and tensile strength by 3.40 in the alloy lacking Boron compared with the alloy containing Boron. Formation of the boride compounds, AlB2, increased the barriers against dislocations and made dislocation more problematic; therefore, this increase of strength was due to the increase and improvement of the precipitate strengthening distribution. On the other hand, thermo-elastic stresses were homogenously and uniformly created in all parts of tensile specimen during controlled quenching

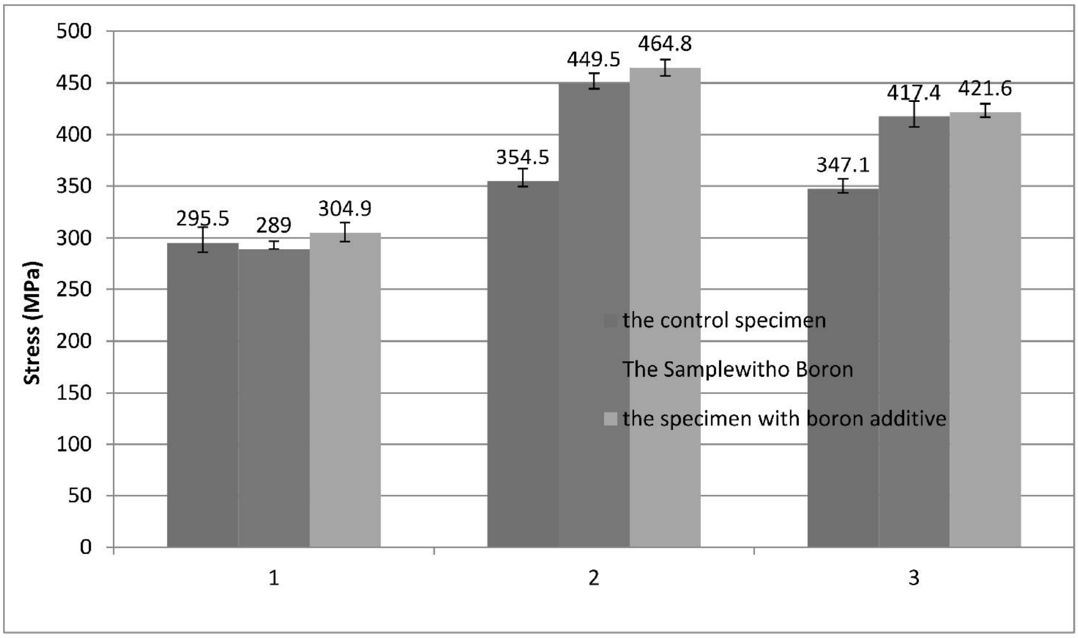

Fig. 8. Comparison of 1) yield stresses (RP0.2 ((MPa), 2) ultimate tensile strength (Rm) (MPa), 3) strength in the fracture point (MPa) of the control specimen (without Boron and with Boron additive), the specimen without Boron additive and the specimen with Boron additive under precipitation hardening operations with controlled quenching for 40 h at aging temperature of 110 ºC(T6C)

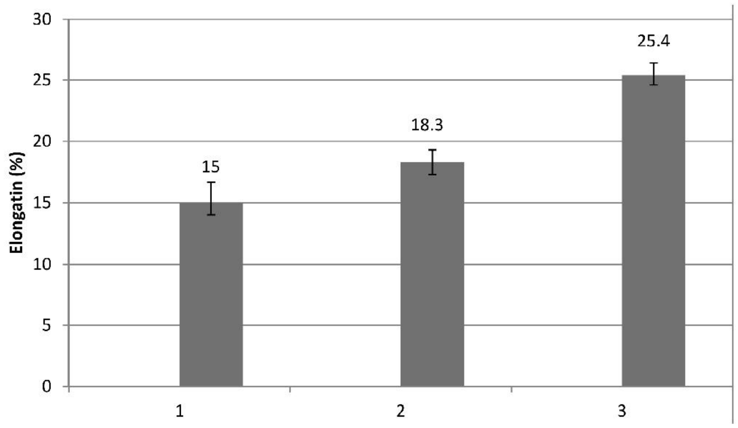

Fig. 9. Elongation comparison of 1) the control specimen (without Boron and without heat treatment), 2) the specimen without Boron additive, 3) the specimen with Boron additive under precipitation hardening operations for 40 h at aging temperature of 110 ºC (T6C)

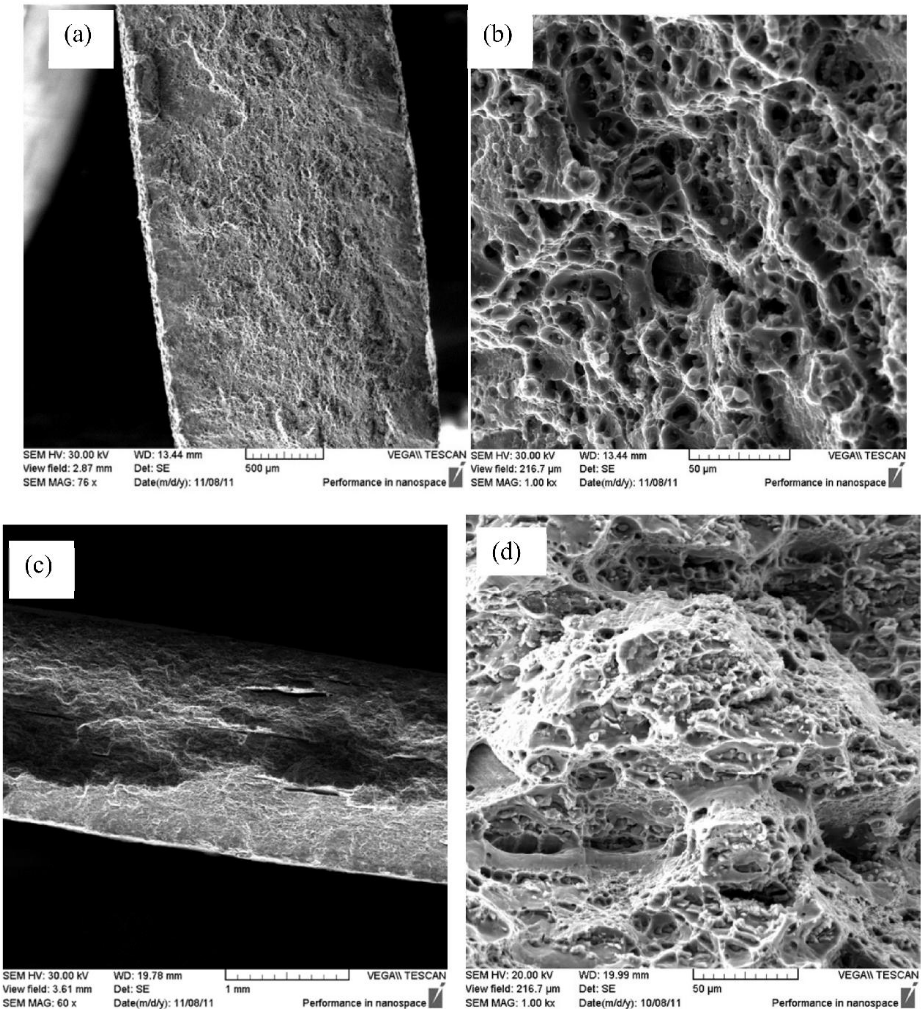

Fig. 10. Fracture cross section of tensile test specimens of 2024 Aluminum alloy a and b) lacking Boron additive , c and d) lacking Boron additive under precipitation hardening operations for 40 min at aging temperature of 110 ºC (T6C) in 1000 times magnification

instead of the hydrostatic compressive stresses. Thus, grain fining of the strengthening precipitates became more uniform [16].

Figure 9 compares fracture strain of the control specimen with the specimen lacking Boron additive and the specimen containing Boron additive.

Boride particles increased ductility by sitting on grain boundaries [17]. Here, location of boride compounds along grain boundaries increased the fracture strain (elongation) of the specimen with Boron by 38.8% compared with the specimen without Boron. Figure 10 compares cross section of the tensile specimens without Boron additive and with Boron additive in different magnifications. These images were taken close to the cross section center of both specimens. In fracture cross section of both specimens, many conical cavities (dimples) were observed. The depth of cavities indicated soft fracture; but, the cross section of the specimen containing Boron had more homogenous transformation and the cavities had more uniform distribution. There were several cracks in the cross section of central area of the specimen (Figure 10c) which were formed by connection of conical cavities and were extended to make a complete rupture. Paying attention to the cross section of this specimen revealed that not only conical cavities were distributed more uniformly, but also there were more secondary phase particles in the cross section.

4. CONCLUSIONS

1. Suitable temperature for concurrent addition of the prepared master alloy mixture to the 2024 Aluminum alloy and alloying was 800 ºC and maintenance time of this mixture inside the melt was 10 min because, at this temperature, free energy of reaction between the molten Aluminum and Boron Oxide was negative. Some resulting Boron from the reduction was dissolved and some precipitated as inter-metallic compounds, which reached suitable growth and distribution at this temperature.
2. Optimal temperature of aging with controlled quenching of 2024 Aluminum alloy containing Boron was 110 ºC due to the suitable distribution and size of the precipitates relating to the aging heat treatment.
3. Boride compounds in 2024 Aluminum alloy acted as heterogeneous grain refining location and caused relative fine-graining of microstructure.
4. The ultimate tensile strength and yield strength at the fracture point of tensile specimens containing Boron were higher than those of the specimens without Boron.
5. The fracture strain (relative elongation percent) of tensile specimens containing Boron increased by 38.80% compared with the specimens without Boron due to the presence of boride particles in grain boundaries.

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