Effect of Heat Treatment on Microstructure and mechanical hardness of aluminum alloy AA7075

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Abstract. The mechanical properties of high strength aluminum alloy AA7075 were greatly affected by heat treatment. This paper was intended to clarify the effect of heat treatment on the microstructure and the mechanical hardness of commercial alloy AA7075-T651. Heat treatment was performed at a variation temperature of 300, 400, 500, and 600°C for 1 h in a vacuum furnace followed by a water quench. The surface observation was done by using optical and electron microscopy and the mechanical hardness was measured by the Vickers Hardness test. The XRD result on as-received alloy revealed the precipitates in the alloy was MgZn$_2$, Al$_7$Cu$_2$Fe, and Al$_2$CuMg. The average hardness of the as-received alloy was 136 HV. The hardness became significantly lower (78.5 HV) after heat treatment at 400°C while heat treatment at others temperature gave hardness in the range 116-122 HV. Heat treatment induced grain growth and dissolution of metastable precipitates. Reduction in the number of MgZn$_2$ and Fe-rich phase decreased the hardness significantly. Heat treatment above 400°C promoted segregation of fresh precipitates which enhanced the metal hardness again. The metallic grain size increased with heat treatment temperature. At 600°C, most of the precipitates were segregated along the equiaxed grain boundaries.

1. Introduction
The high strength aluminum alloy AA7075 has been widely used as a structural material in the aerospace industry. The mechanical characteristic of high strength to weight ratio was obtained by the precipitation hardening mechanism. The alloy composed of alloying element Zn, Mg, and Cu. The intermetallic and strengthening precipitates were formed in the alloy as a result of interaction between the alloying elements and impurities in the alloy [1]. The main precipitates existed in the alloy are the Cu- and Fe-rich phases which include Al$_7$Cu$_2$Fe and (Al, Cu)$_6$(Fe, Cu) with the size in the range of 1-20 µm [1-3]. The strengthening precipitate in the alloy was the MgZn$_2$ phase which segregated as small particles in the order of a few nm at the metallic nanograin boundaries [3]. The other precipitates such as Al$_2$CuMg and Mg$_2$Si were present in small quantities.

The microstructure of AA7075 alloy was sensitive to heat treatment. The heat treatment and aging were reported to change the size, composition, and distribution of the intermetallic precipitates in the alloy [1, 2]. The microstructure change can be monitored by measuring the electrical conductivities and tensile strength. The Cu- and Fe-rich phases exhibited higher potential than that of the Al matrix while the Mg-rich phases are more anodic to the matrix. The presence of nobler elements in the Al tends to enhance the potential of the alloy [4]. Solution heat treatment tended to enhance the potential difference between the intermetallic and the surrounding matrix of AA7075-T6 as proved by the detail studied using electrochemical micro-cell as explained in ref [5] and by a more advanced technique of Scanning
Kelvin Probe (SKP) describe in ref [6]. The change in microstructure in relation to the distribution of the cathodic and anodic intermetallic precipitates can be characterized by measuring the electrical conductivity of the alloy. Solution heat treatment also induced dissolution of the strengthening phase of MgZn$_2$ in the Al matrix which led to the supersaturated solid solution [1]. The supersaturated solid solution of AA7075-T651 was characterized by a higher conductivity relative to the T6 temper due to the high concentration of Mg, Zn, Cu dissolved in the solid solution [3]. The potential gradient between the intermetallic and Al matrix enhanced the susceptibility to galvanic corrosion and pitting [5-7]. The Cu-rich phase, in particular, is detrimental to corrosion attack in Al alloys although present in a small concentration [8].

Other than conductivity tests, the tensile strength measurement was also utilized to monitor the microstructure change in the Al alloy as a result of heat treatment and aging. Heat treatment followed by water quench resulted in a lower mechanical strength of the supersaturated solid solution AA7xxx series than that of the T6 temper [9]. This work is intended to clarify if the alteration in the microstructure as a result of heat treatment can be characterized by measuring the microhardness of the alloy. Heat treatment was conducted on AA7075-T651 at various temperature of 300°, 400°, 500°, and 600°C.

2. Experimental Methods

The material used is a commercial rolled plate AA7075-T651 having a thickness of 2 mm. The plate was cut to give an area of 2 cm$^2$. The specimens were ground from #600 to #1200 grit papers. Finally, the specimens were polished by using an alumina paste to get a mirror finished. Prior to heat treatment, the specimens were cleaned in acetone followed by ethanol in an ultrasonic bath for 3 min each step. Heat treatment was performed at 300, 400, 500, and 600°C for 1 h in a furnace. After heat treatment, the specimens were subsequently quenched in a cold water. To minimize aging, the heat treated specimens were stored in a freezer before further used.

For microstructure observation, the specimens were electropolished in 20% HClO$_4$ solution using 0.25 A for 4 min at 10°C and then degreased in 30% HNO$_3$ solution for 20 s. The microstructure was studied by an optical microscope (Zeiss) and FE-SEM-EDX (FEI INSPECT F-50 & EDAX EDS Analyzer). The crystalline phases existed in the heat treated specimens were analyzed by XRD (Panalytical X’Pert Pro MPD). For measuring the mechanical hardness, Vickers hardness indenter (Struers DuraScan) was utilized by using a 3 kgf load. Five indentations were conducted on each specimen to obtain the average value.

3. Results and Discussion

Figure 1 shows the microstructure development occurred on the AA7075 specimens as a result of heat treatment at 300, 400, 500, and 600°C for 1 h. All of the specimens were priory electropolished to reveal the grain boundaries and the intermetallic precipitates. The as-received specimen and the specimens heat treated below 600°C did not show a clear grain boundary (Figs. 1a-1d). The grain boundaries became clearly observed after heat treatment at 600°C as they were occupied by the segregation of intermetallic precipitates as shown in Fig. 1e.

The as-received alloy was hardened by intermetallic precipitates which included MgZn$_2$, Al$_3$CuMg, and Al$_7$Cu$_2$Fe, pointed by arrows in Fig. 1a. The intermetallic composition was confirmed by EDX analysis. The precipitate Al$_7$Cu$_2$Fe showed an irregular shape while the Al$_3$CuMg and MgZn$_2$ phases tended to have a spherical shape (Fig. 1). In agreement with the reported work in ref [10] that the Al$_7$Cu$_2$Fe, Al$_6$(Cu, Fe) exhibited irregular shape while the Al$_3$CuMg was observed as either irregular or spherically shaped particles. Heat treatment at 300°C induced the formation of fine precipitations as indicated by the numerous black spots in the surrounding larger precipitates (Fig. 1b). The voids which appeared as black spots were formerly occupied by intermetallic which probably detached during electropolishing. The Mg$_5$-rich phase is more anodic to Al [3] and therefore easier to dissolve into the solution during electropolishing. Heat treatment at a higher temperature of 400°C enlarged the intermetallic precipitates size and the surrounding matrix became clearer from small precipitates (Fig. 1e). The big precipitates coarsened in the expense of the smaller ones. Increasing heat treatment...
temperature to 500°C promoted the formation of fresh intermetallic precipitates as indicated by the numerous fine black spots in the perimeter of the bigger precipitates (Fig. 1d). The microstructure of the specimens resulted from heat treatment at 500°C was somewhat similar to that of obtained after heat treatment at 300°C (Fig. 1b). Heat treatment at 600°C led to a significant metallic grain growth with an equiaxed shape as can be seen in Fig. 1e. The recrystallization of metallic grain to result in an equiaxed grain type was only obtained in the AA7075 alloy as a result of heat treatment at a high temperature of 600°C [10]. The grain size was in the range of 100-200 µm. The Cu-rich intermetallic precipitates were segregated along the grain boundaries forming a nearly continuous network. The grain bodies were relatively free from precipitates as shown in Fig. 1e.

The coarsening of the microstructure as a result of heat treatment in the AA7075 specimens followed the Ostwald ripening mechanism where the larger precipitates grew at the expense of the small one [11]. The driving force for such a phenomenon is the gradient of solubility of the solute atom (Cu and Zn) between the big and small precipitates. At an increasing temperature, the solute atoms move from the small to the big precipitates by a diffusion mechanism.

![Figure 1. FE-SEM images of the AA7075 specimens showing the microstructure of a) as-received, and after heat treatment at b) 300°C, c) 400°C, d) 500°C, and e) 600°C.](image)

Confirming the EDX analysis results, Figure 2 presents the XRD pattern of AA7075 after heat treatment at 300, 400, 500, and 600°C showing the phases existed in the alloys in comparison to the as-received one. All of the curves showed a similar serial peaks position for the Al matrix phase at the angles of 38.4°, 44.6°, 64.9°, 78.1°, and 82.4°. In addition to the Al phase, the as-received specimen composed of hardening precipitates MgZn$_2$, Al$_2$CuMg, and Al$_2$Cu$_2$Fe as indicated by the small peaks in between the angle 15°-85° in the XRD pattern. The peaks for Al$_2$Cu$_2$Fe phase at the angles of 39.0, 65.0, 78.2° and 82.6° were slightly overlapped with the peaks for Al. The MgZn$_2$, Al$_2$CuMg, and Al$_2$Cu$_2$Fe phases remained in the alloy after the heat treatment of 300, 400, and 500°C. However, the specimen heat-treated at 400°C showed more peaks for Al$_2$CuMg especially at low angles between 15° and 30°. The corresponding peaks did not exist in the specimen heat-treated at 300° and 500°. In agreement to the microstructure observation, heat treatment at 300 and 500°C induced the growth of MgZn$_2$ and Al$_2$Cu$_2$Fe precipitate while oppositely heat treatment at 400°C enlarged the Al$_2$CuMg precipitate. The peaks for MgZn$_2$ and Al$_2$Cu$_2$Fe phases partly disappeared for the specimen heat-treated at 400°C. Heat treatment at the highest temperature of 600°C left mainly MgZn$_2$ and Al$_2$Cu$_2$Fe precipitates as shown in
the Fig. 2. The result which confirmed the FE-SEM and EDX results shown in Fig. 1. The Al$_2$CuMg phase was no longer detected at the specimen heated at 600°C.

![Figure 2](image-url)  
**Figure 2.** The XRD pattern of AA7075-T651 after heat treatment at 300, 400, 500, and 600°C for 1 h.

![Figure 3](image-url)  
**Figure 3.** Mechanical hardness of AA7075-T651 after heat treatment at 300, 400, 500, and 600°C for 1 h.

Figure 3 shows the effect of heat treatment on the mechanical hardness of AA7075 specimens. The as-received alloy exhibited an average hardness of 136 HV. In general, the hardness tended to be lower as a result of heat treatment. Similar to the reported [1, 2, 9] investigation on the effect of solution heat treatment, the microhardness decreased as a result of heat treatment. The specimens which heat-treated at 300 and 500°C had a nearly similar hardness of 121.4 and 122.4 HV, respectively, as expected since they exhibited similar microstructure (Figs. 1 and 2). Heat treatment at 400°C resulted in the lowest hardness of 78.5 HV which was nearly half of the as-received specimens. Significant reduction in the number of MgZn$_2$ and Fe-rich precipitate was accused to be the reason for the corresponding hardness depression. The specimen which was heat treated at 600°C exhibited a hardness of 116.2 HV which was slightly lower than that of heat-treated at 300 and 500°C. The hardening precipitates were not distributed
in the metallic grain as a result of heat treatment at 600°C but aggregated as a continuous precipitate along the grain boundaries.

4. Conclusion
The effect of heat treatment on the microstructure of AA7075 alloy has been investigated. The results showed that heat treatment at temperature 300-600°C gave a significant impact on the microstructure transformation and the resulting mechanical hardness of the alloy. A slight reduction (~15 HV) in the hardness was obtained as a result of heat treatment at 300°C and 500°C. At both temperatures, the Fe-rich precipitates grew larger while the Mg-rich precipitates became smaller following the Ostwald ripening mechanism. Oppositely at 400°C, the dominant precipitate observed in the alloy was the Mg-rich phase which formed a big particle of about 10 µm while the matrix was relatively clean from small precipitates. Such microstructure resulted in a low hardness of 78.5 HV. A significant different microstructure was attained as a result of heat treatment at 600°C where the Cu-rich precipitates segregated along the grain boundaries forming a nearly continuous layer while the grain interior was relatively free from precipitates. The segregation of precipitates along grain boundaries did not give a significant contribution on the hardness of the bulk specimen and therefore the hardness was slightly lower (116.2 HV) than that of resulted from 300°C and 500°C heat treatment (121-122 HV). The results indicated that the transformation in the microstructure of AA7075 alloy can be characterized by measuring the microhardness of the alloy.

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