

Influence of Heat Treatment on the Mechanical Properties of AA6066 Alloy

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Abstract

The microstructural and mechanical characterization of heat treatable 6xxx (Al-Mg-Si-Cu based) wrought aluminum alloys was studied. The aim of this work was to produce a fine grained, high strength 6xxx series aluminum alloy by adjusting the processing conditions, namely deformation, solutionizing and aging. Their effects were investigated in terms of microstructure using SEM analysis and mechanical properties by tensile tests and hardness measurements. The initial characterizations showed that Mg_2Si and $(Fe,Mn,Cu)_3SiAl_{12}$ were the primary particles observed in the α -Al matrix. Nearly 140HB hardness was obtained with solutionizing at 530 °C and aging at 175 °C for 8 h, which was the optimum treatment for obtaining peak hardness. When shaping (deformation) was concerned, 10% swaging before solutionizing yielded lower strength and hardness as compared to the 40% swaging due to lower strains finalizing partial recrystallization in the 10% swaged specimens.

Key words: Al alloy 6xxx series, Deformation, Grain size, Microstructural characterization.

Introduction

Aluminum alloys have been the material of choice for aircraft construction since the 1930s. The aerospace industry relies heavily on 2xxx and 7xxx alloys, while 6xxx aluminum alloys are of particular interest nowadays. According to Troeger (2000), 6xxx alloys have numerous benefits including medium strength, formability, weldability, corrosion resistance, and low cost. He states that 6xxx can be used in a variety of applications including aircraft fuselage skins and automobile body panels and bumpers, instead of more expensive 2xxx and 7xxx alloys, after appropriate heat treatments. Hence, microstructural characterization of the alloy and processing procedure is important for that approach.

Characterization of Al-6xxx alloys has been the subject of many studies. It is known that the main components of heat treatable 6xxx series Al alloy are Mg and Si, and 6xxx derives its strength from

the precipitation hardening phase, Mg_2Si . The volume fraction of Mg_2Si is affected primarily through the level of Mg within the alloy, but the Si content is also important. Hirth et al. (2001) concluded that increasing Si in 6xxx type alloys increases strength in the T4 and T6 tempers. Another study on 6xxx carried out by Matsuda et al. (2002) showed that the addition of copper to Al-Mg-Si alloys not only changes the precipitation sequence but also enhances hardness and refines microstructure by segregating to the $Q' \setminus \alpha - Al$ interface. In addition to Mg-Si-Cu, manganese and chromium are also used as alloying elements. Dorward and Bouvier (1998) explained the beneficial effects of manganese and chromium, namely that these alloying additions inhibit the precipitation of magnesium and silicon on grain boundaries, thereby reducing intergranular fracture tendencies. The beneficial effect of manganese (or other incoherent dispersoid-forming elements) on toughness is homogenization of deformation, leading to a

reduction in intergranular fracture. In addition, they noted that a refined grain size, as influenced by manganese (a grain structure control agent), was another positive effect.

According to the alloy composition and aging procedure, different precipitates can be observed in the microstructure, which will affect the final mechanical properties. Chakrabarti and Laughlin (2004) tried to place some of these precipitates on a phase diagram. By using their line diagram, the stable phases at RT can be observed.

In order to obtain optimum mechanical properties, a suitable production procedure should be selected. In addition to aging time and temperature, the presence of deformation, its temperature and place in the whole production procedure should be carefully checked. Zhen et al. (1997) stress the importance of time passed between solution heat treatment and artificial aging, which shows that any time spent between quenching and aging leads to natural aging of the specimen and hence lowers hardness in the end. In another study, Sun et al. (1999) deformed samples between quenching and aging and they concluded that this method has positive effects on hardness and strength. A different method was used by Cai et al. (2004). They tried a dynamic aging procedure (integrated process combining thermo-mechanical processing and aging). In summary, dynamic aging was superior to conventional aging in terms of both mechanical properties and time to peak strength.

All studies on 6xxx above were based on Al-6061 and 6063 type alloys. In our work, the properties of a 6xxx alloy containing Si, Mg, Cu and Mn in the order of 1wt% conforming to AA6066 aluminum alloy were investigated. Al-6063 has balanced Mg-Si to form stoichiometric Mg_2Si and Al-6061 has a Cu ingredient besides balanced Mg-Si; on the other hand, Al-6066 has both Cu and excess Si. As stated previously, different properties were obtained with various amounts of alloying elements. Hence, the aim of the present study was to optimize the heat treat-

ment and to investigate the effect of initial deformation (shaping) process on the mechanical properties of Al-6066 type alloy.

Experimental Procedure

This research was conducted using wrought 6xxx series aluminum rods with the dimensions of $\varnothing = 46$ mm and $l = 200$ mm. The as-received materials with the chemical composition stated in Table 1 were in the annealed temper, which conforms to AA6066 alloy.

The microstructural investigations and the mechanical testing were carried out as an initial characterization step for the 3 tempers: annealed, solutionized-water quenched (WQ) and aged.

The effect of temperature and time during solutionizing and aging were examined as the second step. For these experiments, the rods were cut into 40 equal slices. Throughout the study same-dimensioned test specimens were used. In order to investigate the effect of solutionizing temperature, the samples were solutionized for 95 min and for 12 h at 4 different temperatures (515, 530, 540 and 550 °C). A 95 min duration was calculated according to the dimensions of specimens suggested by the Aluminum Association. Twelve hours was selected arbitrarily to investigate the effect of soaking time on hardness. All solutionizing treatments were performed in a muffle furnace operating with ± 1 °C. Temperature of the furnace was controlled by an extra secondary thermocouple. Neither air nor gas circulation was used in the furnace.

After solutionizing, all samples were quenched in water at room temperature. Ice was used to keep the temperature at about 20 ± 2 °C. During quenching, the medium was stirred to achieve temperature homogeneity. It must be noted that, after solutionizing treatment, the samples were always kept in a freezer, which was kept at about -18 °C to eliminate the detrimental effect of natural aging.

Table 1. The nominal chemical composition of the alloy used.

Si %	Mg %	Cu %	Mn %	Fe %	Ti %	Zn %	Ni, Cr, Pb, Sn, Ca, Sr	Al %
1.21	0.92	0.82	0.65	0.26	0.013	0.010	≈ 0	balance

For the ideal solutionizing temperature, 24 different sets of aging trials were carried out. The selected aging temperatures were 150, 175 and 200 °C. The specimens were held at these temperatures for 2 to 16 h with a 2-h interval. The aging treatment was carried in a Julabo heating circulator oil bath, which operates with 0.1 °C accuracy.

As a final step, the effect of the swaging process prior to heat treatment was investigated. Rods were machined with 4 different diameters such that approximately 10%, 20%, 30% and 40% deformation (reduction in area) could be established. The deformation process was done with the use of a Fenn rotary die swager at room temperature.

The detailed processing of these specimens is tabulated in Table 2.

For microstructural analysis, specimens were ground with emery papers from 600 to 1200, and polished with 1 μm diamond paste. As an etchant, a mixture of HF, H₂SO₄ and H₂O in the composition 1:2:17 was used. For characterization, optical microscopy and a scanning electron microscope (JEOL JSM 6400® equipped with Noran® EDS system) were used. Image analysis was carried out

with Clemex Vision Professional software.

For hardness measurements, Brinell hardness numbers were obtained with a 2.5 mm ball indenter under 613 N load. For tension testing, samples were prepared according to the aluminum tensile testing standard of ASTM (B557M-02a).

Results and Discussion

Microstructural Features and Mechanical Properties

As seen in Figure 1, the backscatter SEM images and optic micrographs on as-polished surfaces revealed 2 types of particles in the α -Al matrix: large black particles and tiny (5-10 μm) gray script-like features. With the assistance of EDS analysis (Figure 2), the features were possibly identified as Mg₂Si and (Fe,Mn,Cu)₃SiAl₁₂.

Highly alloyed 6xxx had complex intermetallics originating from cast ingots. Since iron was the omnipresent impurity element and had a very low solubility in aluminum, iron-rich phases could be seen in all aluminum alloys. The presence of manganese, chromium or copper leads to the formation of

Table 2. Processing schedule.

#	DEFN %	SOLN		AGING	
		Temp (°C)	Time (min)	Temp (°C)	Time (h)
1		-	-	-	-
2		515			
3	-	530			
4		540	95, 720	175	8
5		550			
6				150	
7	-	530	95	175	2, 4, 6, 8, 10, 12
8				200	
9	10				
10	20				
11	30	-	-	-	-
12	40				
13	10				
14	20				
15	30	530	95	-	-
16	40				
17	10				
18	20				
19	30	530	95	175	4, 6, 8, 10, 12
20	40				

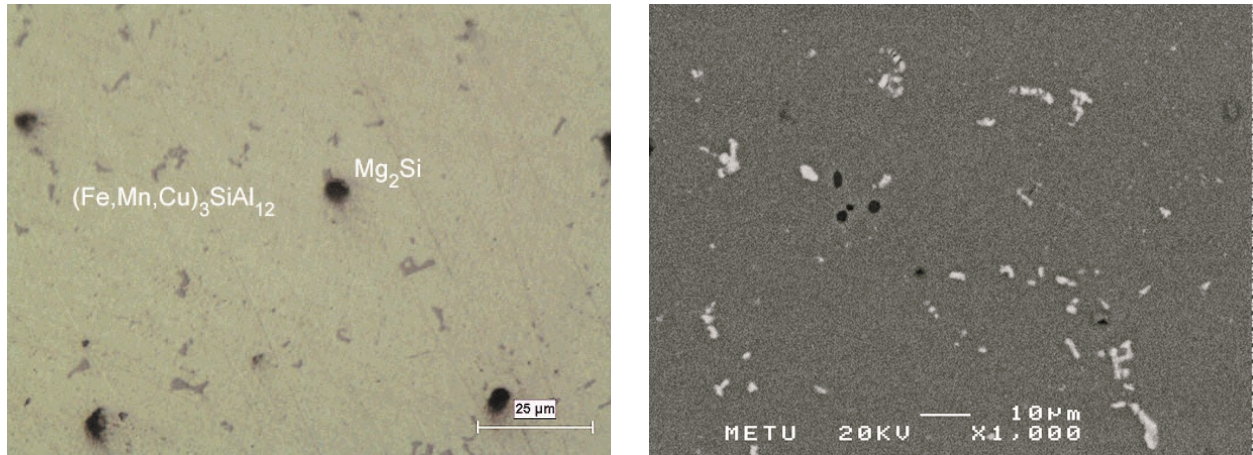


Figure 1. (a) Optical micrograph and (b) backscatter SEM image of as-polished surface.

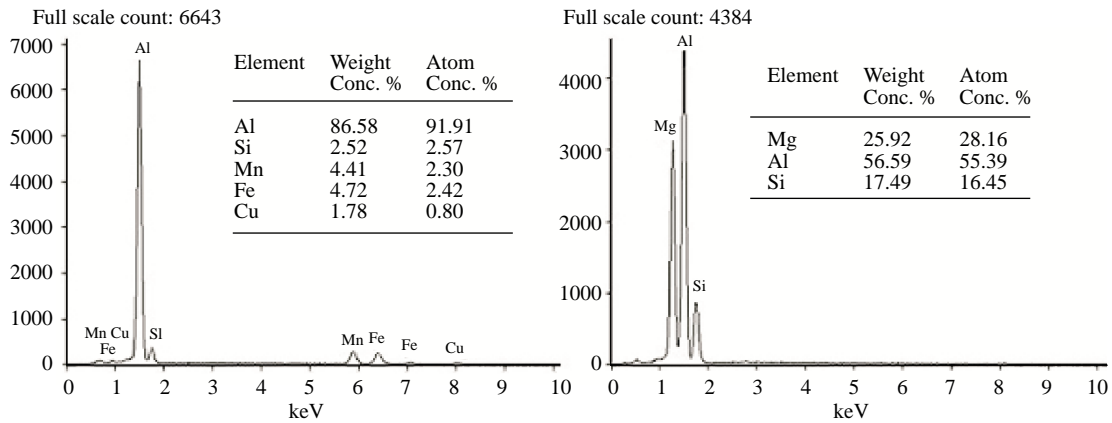


Figure 2. EDS analysis of microscopic features.

Table 3. Mechanical test results for O, T4 and T6 tempers and their comparison with the standard AA6066.

	Experimental Results			Literature ASM (1996)		
	Brinell Hardness	Yield (MPa)	UTS (MPa)	Brinell Hardness	Yield (MPa)	UTS (MPa)
6066 - O	59-60	168-181	220-223	43	82.7	152
6066 - T4	100-103	300-335	442-445	90	207	359
6066 - T6	128-140	441-461	461-478	120	359	393

$(\text{Fe,Mn,Cu})_3\text{SiAl}_{12}$. The other phase, Mg_2Si , was the main ingredient of δ xxx, which would readily dissolve during solutionizing and contribute to the precipitation hardening for the period of artificial aging.

The mechanical properties of the alloys at the 3 tempers are summarized in Table 3. It is evident that the alloy used in the study fulfilled the minimum requirements given by the Aluminum Association.

Ideal Heat Treatment Cycle

Solutionizing is the main step for precipitation hardening, and it must be carried out carefully in order to avoid grain boundary melting due to overheating. The effect of solutionizing treatment is summarized via a column chart in Figure 3. The as-received specimen in the annealed temper having a hardness of 57HB was treated with different temperatures and times before artificial aging at 175 °C for 8 h. Peak

hardness was achieved after solutionizing at 530 °C for 12 h. The chart demonstrated that, as the temperature increased for 95 min solutionizing, the hardness increased from 131 to 144. Actually this was foregone; as temperature increases more solid dissolves and mixes in the matrix for super saturation, due to increased diffusion kinetics and higher solubility limits at higher temperatures. Another output of the chart could be on the soaking times. Below 530 °C, there was a tendency towards increasing hardness with increasing soaking time, whereas the trend was the opposite for temperatures above. Below 530 °C, with the increase in soaking time, a satisfactory degree of solution of the undissolved or precipitated soluble phase constituents, forming a good homogeneity of solid solution, could be established. After 530 °C, the loss of hardness for long soaking times might be interrelated with the most probable grain growth tendency. Although grain boundary melting was not observed, to be on the safe side 95 min duration at 530 °C could be chosen as the solutionizing treatment for the alloy since the hardness values were closer to each other.

The aging behavior of the alloy is summarized in Figure 4. It shows typical hardness curves of solutionized alloys after artificial aging at elevated temperatures. A peak hardness of 139 HB could be attained after 8 h of aging at 175 °C and then the hardness decreased slightly. The curve for 200 °C did not show peak hardness but it could be predicted to be in the vicinity of a half an hour or so. The quick drop in the curve was an indication of the fast overaging

due to high diffusion rates at high temperatures. On the other hand, the effect of slow kinetics could be the conclusion of the aging at 150 °C. The hardness value reached after 16 h could be obtained by aging at 175 °C for 2 h only. Hence it can be concluded from Figure 4 that aging at 175 °C was the optimum for the alloy, since it yielded the desired hardness values of 130-140HB in 2-16 h.

Effect of Deformation

The 6xxx type of aluminum alloy is used for general extrusion purposes. Hence the effect of deformation behavior prior to heat treatment must be considered. Ultimate tensile strength (UTS) and 0.2% offset yield strength of non-deformed and deformed samples after peak aging were obtained from the stress-strain diagram and are summarized in Table 4. At first sight, deformed specimens had lower strength as compared to non-deformed ones. The only reason for that would be the larger grain sizes in the deformed samples as compared to the original non-deformed ones having a mean grain size of $18 \pm 4.77 \mu\text{m}$. The effect of deformation on final grain size is shown in Figure 5. Another output of the mechanical test was the increase in strength with an increase in percent deformation. Deformation leads to energy storage for the period of lattice defect creation, i.e. dislocations. During solutionizing at temperatures of 530 °C, the cold-deformed specimens led to recrystallization. It was known that the higher the strains, the lower the recrystallized grain size. Hence, after

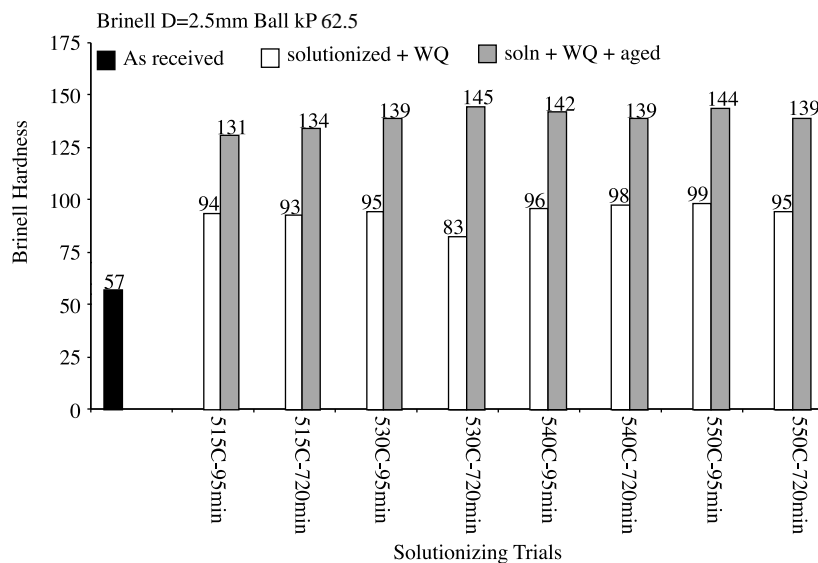


Figure 3. Effect of solutionizing treatment on hardness prior to aging at 175 °C for 8 h.

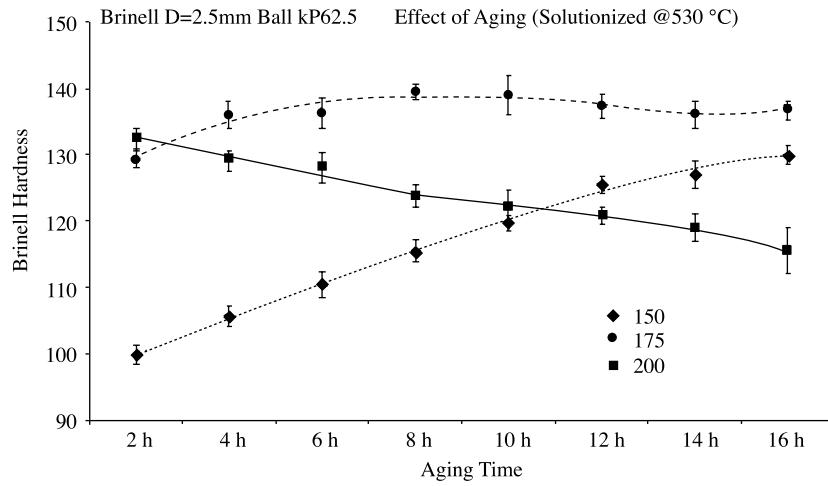


Figure 4. Comparison of aging behavior of solutionized alloy aged at temperatures of 150, 175 and 200 °C.

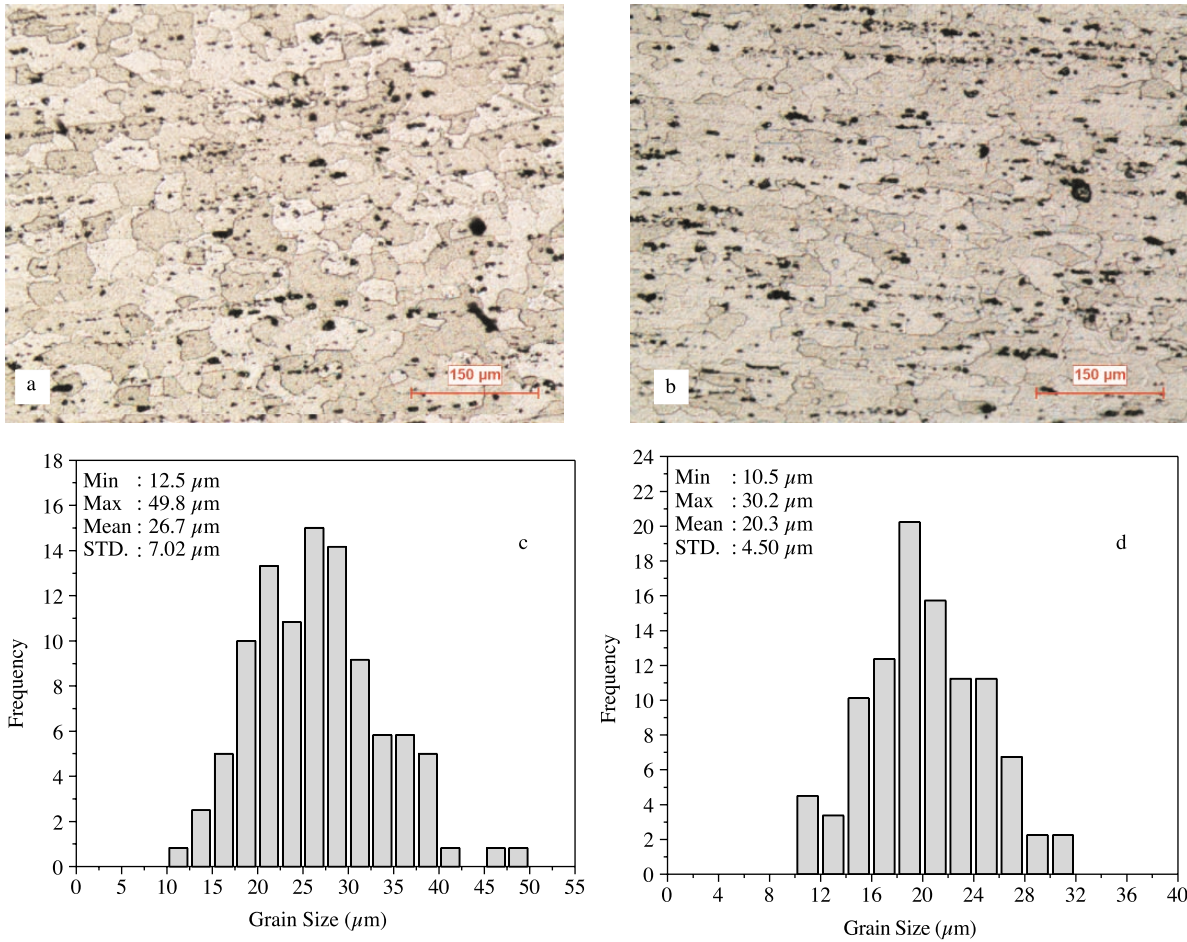


Figure 5. Optic micrographs after solutionizing and the correspondence grain size distributions; a, c) 10% deformed samples; b, d) 40% deformed samples.

solutionizing the 10% deformed sample with mean grain sizes in the order of $26.7 \pm 7.02 \mu\text{m}$ had UTS of 376 MPa, where the 40% deformed sample with $20.5 \pm 4.72 \mu\text{m}$ average grain size had UTS of 418 MPa.

Table 4. Tensile strength of AA6066 alloy solutionized at 530 °C and peak-aged at 175 °C for 8 h after deformation.

Deformation Status	UTS	Yield Strength
<i>Non-deformed</i>	470	450
<i>10% RT</i>	376	295
<i>20% RT</i>	380	298
<i>30% RT</i>	383	300
<i>40% RT</i>	418	319

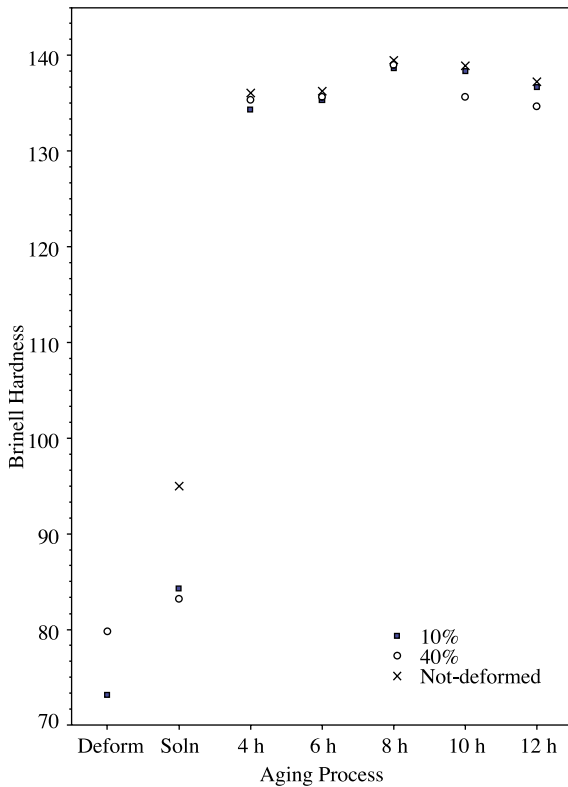


Figure 6. Effect of deformation on the hardness of alloy.

A similar trend could be obtained from hardness values. The variations in hardness for non-deformed,

10% and 40% deformed specimens are presented in Figure 6. Right after deformation, the 40% deformed specimen had 79HB due to higher dislocation density than the 10% deformed one with 73HB. After recrystallization during solutionizing and further artificial aging, a hardness trend similar to that in UTS was observed.

Conclusion

In this study, the effect of deformation on mechanical properties of a 6xxx series aluminum alloy was investigated. Following the determination of the ideal conditions for solutionizing and aging processes, specimens were mechanically deformed by swaging at 4 different deformations and then heat treated. The primary conclusions obtained from this study can be summarized as follows:

1. Two types of particles were observed in the alloy: black Mg_2Si and gray script-like $(\text{Fe,Mn,Cu})_3\text{SiAl}_{12}$, which were equilibrium constituents coming from the cast ingot.
2. The ideal solutionizing temperature was 530 °C. Below 530 °C, there was a tendency towards increasing hardness with increasing soaking time, whereas the trend was the opposite for temperatures above.
3. Aging trials between 150 and 200 °C showed that peak hardness values could be obtained after aging at 175 °C for 8 h. As compared to the fast overaging at 200 °C and slow hardening at 150 °C, 175 °C was confirmed as the optimum aging temperature for industrial usage.
4. As deformation amount increased, the recrystallized grain size got smaller, enhancing the strength and hardness.

Acknowledgment

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References

- ASM Specialty Handbook, "Aluminum and Aluminum Alloys", Materials Information Society, 1996.
- ASTM Standard, "B557M Tension Testing Wrought and Cast Aluminum and Magnesium-Alloy Products", 2002.
- Cai, M., Field D.P. and Lorimer G.W., "A Systematic Comparison of Static and Dynamic Ageing of Two Al-Mg-Si Alloys", Materials Science and Engineering, A373, 65-71, 2004.
- Chakrabarti, D.J. and Laughlin, D.E., "Phase Relations and Precipitation in Al-Mg-Si Alloys with Cu Additions", Progress in Materials Science, 49, 389-410, 2004.
- Dorward, R.C. and Bouvier, C., "A Rationalization of Factors Affecting Strength, Ductility and Toughness of AA6061-Type Al-Mg-Si-(Cu) Alloys", Materials Science and Engineering, A254, 33-44, 1998.
- Hirth, S.M., Marshall, G.J., Court, S.A. and Lloyd, D.J., "Effects of Si on the Aging Behaviour and Formability of Aluminium Alloys Based on AA6016", Materials Science and Engineering, A319-321, 452-456, 2001.
- Matsuda, K., Teguri, D., Uetani, Y., Sato, T. and Ikeno, S., "Cu-Segregation at the Q'/ α -Al Interface in Al-Mg-Si-Cu Alloy", Scripta Materialia, 47, 833-837, 2002.
- Ravi, C. and Wolverton, C., "First-Principles Study of Crystal Structure and Stability of Al-Mg-Si-(Cu) Precipitates", Acta Materialia, 52, 4213-4227, 2004.
- Singh, R.K., Singh, A.K. and Prasad, N.E., "Texture and Mechanical Property Anisotropy in an Al-Mg-Si-Cu Alloy", Materials Science and Engineering, A277, 114-122, 2000.
- Sun, Y., Baydoğan, M. and Çimenoglu, H., "The Effect of Deformation Before Ageing on the Wear Resistance of an Aluminium Alloy", Materials Letters, 38, 221-226, 1999.
- Troeger, L.P. and Starke, E.A., "Microstructural and Mechanical Characterization of a Superplastic 6xxx Aluminum Alloy", Materials Science and Engineering, A277, 102-113, 2000.
- Zehn, L., Kang, S.B. and Kim, H.W., "Effect of Natural Aging and Preaging on Subsequent Precipitation Process of an Al-Mg-Si Alloy with High Excess Silicon", Materials Science and Technology, 13, 905, 1997.