

Significant Increase in Strength and Elongation of Al-3.7Cu-1Mg Alloy via Short Age-Treatment Cycle

B. Mirzakhani^{1,2}, Y. Payandeh¹, H. Talebi¹ and M. Maleki¹

* b-mirzakhani@araku.ac.ir

Received: January 2020

Revised: April 2020

Accepted: May 2020

¹ Department of Materials Science and Engineering, Faculty of Engineering, Arak University, Arak 38156-8-8349, Iran.

² School of Metallurgy and Materials Engineering, Iran University of Science and Technology, Tehran, Iran.

DOI: 10.22068/ijmse.17.3.30

Abstract: In this paper, the effect of two-step precipitation hardening on the mechanical properties of Al-3.7Cu-1Mg was investigated. For this meaning, some specimens were subjected to the first step aging at 175, 190 and 205 °C for 2 h, once the samples solution treated at 500 °C. To have stable precipitates uniformly distributed in the microstructure and to reduce the heat treatment time, the second step was implied at 65 °C. The tensile and hardness tests were performed at ambient temperature immediately after aging. The results indicated that depending on the first step temperature, the second aging time affects the mechanical behavior of the alloy in different aspects. A factor named SNMP introduced to determine the cycle giving the best mechanical properties. The strength and elongation increase 1.5 and 2 times, respectively; compared to the values reported in the DIN EN 755-2 standard by performing the two-step aging cycle, consisting of the first-stage at 175 °C and the second step at 65 °C for 10 hours. Moreover, using the proposed two-step aging, the heat treatment time was reduced considerably compared to the conventional precipitation-hardening process.

Keywords: Aluminum alloy, Two-step aging, Mechanical properties, SNMP, Aging time.

1. INTRODUCTION

Aluminum alloys have been considered as an alternative to steels in recent years. Thanks to the high strength-to-weight ratio, recyclability, and good formability, aluminum alloys are widely used in different industries such as automotive and aerospace for economic and environmental reasons. To properly use such materials, it is important to study the production conditions, including heat treatment. Aging is a common method used to improve the strength of heat treatable aluminum alloys, such as Al-Cu series alloys. It is necessary to control the heat treatment conditions, to obtain the expected properties. Researchers have proposed artificial aging for some of these series alloys [1, 2]. Studies show that T3, T4, T3510, T3511, T4511, T4510 tempers are generally used for Al-3.7Cu-1Mg alloy in the industries. According to the DIN EN 755_2 standards [3, 4], the mechanical properties given in Table 1 achieved by implementing these natural aging cycles. Also, artificial aging including T6, T8, T616, T618 tempers were proposed for some 2000 series alloy [5]. Researchers have used

two-step aging for heat treatable alloy as well. Double precipitation hardening is a technique to make a good distribution of precipitates at low-temperature treatment after first step aging at high temperature [6]. Due to this heat treatment process, the mechanical properties change, which was first reported by Löffler et al. [7] for Al-Zn alloys. The treatment consists of natural aging after the first step aging at 180 °C. Lumley et al. [8, 9] suggested that the two-stage artificial aging for at least 7 days is required for the improvement of mechanical properties of heat treatable aluminum alloys. It has been reported that secondary precipitates could form during the second step aging at 25-65 °C once the first step is carried out at 150 °C [10, 11]. This temper (known as T614), results in fine and distributed precipitates due to the second step aging, which could simultaneously increase the strength and toughness of aluminum alloys [12-15].

Studies show that the interrupted precipitation hardening leads to improvement in mechanical properties of some 2000 series alloys, but these heat treatment cycles are too long (7-14 days) [8, 9]. Although double artificial aging has been utilized for a few Al-Cu alloys [12].

Table 1. The mechanical properties of the Al-3.7Cu-1Mg alloy, listed in standard (DIN EN 755-2) [3, 16]

| Hardness (HB) | Elongation (%) | Tensile Strength (MPa) | Yield Strength (MPa) |
|---------------|----------------|------------------------|----------------------|
| 115 | 8 | 370 | 250 |

Table 2. Chemical composition of Al-3.7Cu-1Mg alloy used in this work (wt %)

| Al | Pb | Ti | Zn | Cr | Mg | Mn | Cu | Fe | Si |
|------|------|------|------|------|------|------|------|------|------|
| Bal. | 1.00 | 0.01 | 0.26 | 0.04 | 0.98 | 0.33 | 3.68 | 0.41 | 0.32 |

This treatment has not been implemented for Al-Cu-Mg alloy. In this work, the effects of single and double aging on the tensile properties and hardness of Al-3.7Cu-1Mg alloy were investigated to improve the strength, ductility, and hardness of standard value. Moreover, the total aging time was considered as a critical factor in designated heat treatment cycles which is interesting for industrial applications.

2. EXPERIMENTAL PROCEDURE

In the present study, the hot-extruded bar of Al-3.7Cu-1Mg alloy with the chemical composition listed in table 2 was used. The samples cut from the primary bar and were solutionized at 500 °C for 1 hour. They were then initially aged at temperatures of 175, 190, and 205 °C for 2 hours, water quenched, and finally aged in the second step at room temperature and 65 °C for 10, 50, and 100 hours (Fig.1). The single and double precipitation hardening conditions and related sample codes are listed in table 3.

The tensile samples with 45 mm gauge length and 9 mm diameter were tested according to E8/E8M standard using a Santam-STM 150 machine at ambient temperature with a displacement rate of 1 mm/min. The Brinell hardness measurement

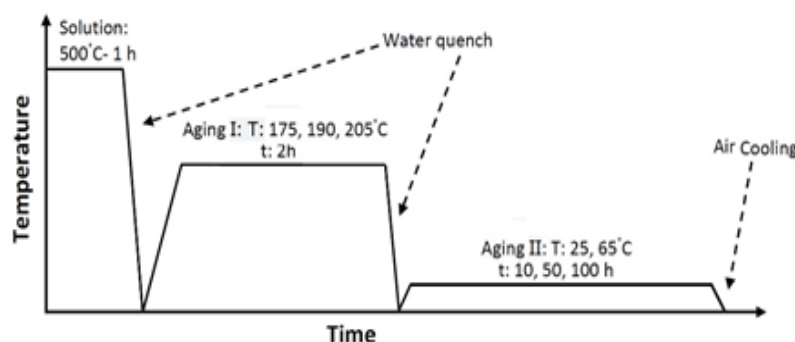
was also done according to the ISO/6506-1 standard with 2.5 mm steel ball and 6.25 kgf load. To study the microstructure, the samples were examined by optical microscopy and TESCAN VEGA//XMU scanning electron microscopy. To reveal microstructure the etchant of 36 mL HCl-45 mLHNO₃-3 mL HF-43 mL H₂O-12 g Chromic acid was used at room temperature for 5 s.

3. RESULTS AND DISCUSSION

Mechanical behavior and microstructure of the samples at different step of heat treatment cycles (Fig1) have been compared and analyzed.

3.1. Single Aging

Fig .2 shows the stress-strain curves of the samples after the first step aging at various temperatures. As shown in this Fig, increasing temperature increases the yield and tensile strength and then decreases for the samples aged at 205 °C. On the contrary, when the temperature rises from 175 to 190 °C, the elongation increases and then decreases by a further increase in temperature to 205 °C. Indeed, by aging at 190 °C the volume fraction of precipitates increases compared to the sample aged at 175 °C; therefore,

**Fig. 1.** Heat treatment cycles of precipitation hardening used in this work

the strength increases. However, dislocations lock by fine precipitates and the elongation of the material reduces. Further increase in aging temperature causes the precipitates to grow up and material undergoes over age. So the average distance traveled by the dislocation raises and strength decreases in comparison to the sample treated at 190 °C. It could be concluded that the single age process carried out at 190 °C results in the improvement of strength significantly and the treatment at 175 °C leads to the highest elongation.

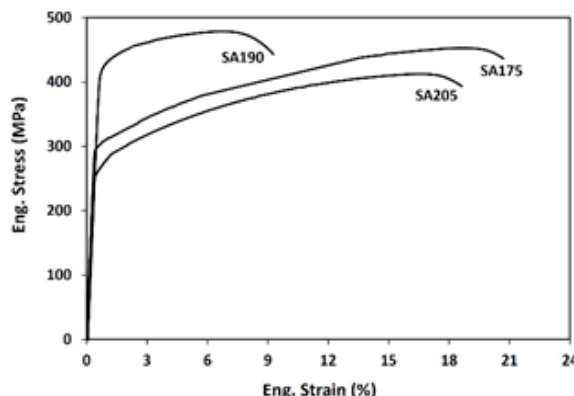


Fig. 2. Stress-strain curves of samples single aged at temperatures of 175, 190, and 205°C for 2h.

3.2. Double Aging

Fig.3 displays the tensile test results of the samples, initially age hardened at 175, 190 and 205 °C for 2h and then in the second step subjected to precipitation hardening at 65 °C for different . The plan of interrupted aging is that

the instability of precipitates formed in the first step of aging causes changes in their size, volume fraction and morphology during the second step aging cycle . Therefore the mechanical properties of material modify as seen in Fig. 3. Generally, short artificial aging in the second

Table 3. Single and double aging conditions and related sample codes.

| Single/ Double aging | Aging temperature and time | Samples code |
|--------------------------------------|--------------------------------------|--------------|
| Single aging | 175°C-2h | SA175 |
| | 190°C-2h | SA190 |
| | 205°C-2h | SA205 |
| Double aging | Aging I:175°C-2h, Aging II:25°C-10h | DA1752510 |
| | Aging I:175°C-2h, Aging II:25°C-50h | DA1752550 |
| | Aging I:175°C-2h, Aging II:25°C-100h | DA17525110 |
| | Aging I:175°C-2h, Aging II:65°C-10h | DA1756510 |
| | Aging I:175°C-2h, Aging II:65°C-50h | DA1756550 |
| | Aging I:175°C-2h, Aging II:65°C-100h | DA17565100 |
| | Aging I:190°C-2h, Aging II:25°C-10h | DA1952510 |
| | Aging I:190°C-2h, Aging II:25°C-50h | DA1952550 |
| | Aging I:190°C-2h, Aging II:25°C-100h | DA19525100 |
| | Aging I:190°C-2h, Aging II:65°C-10h | DA1956510 |
| | Aging I:190°C-2h, Aging II:65°C-50h | DA1956550 |
| | Aging I:190°C-2h, Aging II:65°C-100h | DA19565100 |
| | Aging I:205°C-2h, Aging II:25°C-10h | DA2052510 |
| | Aging I:205°C-2h, Aging II:25°C-50h | DA2052550 |
| | Aging I:205°C-2h, Aging II:25°C-100h | DA20525100 |
| Aging I:205°C-2h, Aging II:65°C-10h | DA2056510 | |
| Aging I:205°C-2h, Aging II:65°C-50h | DA2056550 | |
| Aging I:205°C-2h, Aging II:65°C-100h | DA20565100 | |

step improves the tensile properties of the alloy. As illustrated in Fig.3-a, Fig.3-b and Fig.3-c, the strength increases after 10 h aging and then decreases once the holding time increases to 50 and 100 h. It could be attributed to the fact that during the second step of aging at 65 °C, precipitates formed in first step aging, could be distributed as clusters and reach the stable state after about 10 h. These cluster-like precipitates cause the dislocation glide to become difficult and the strength to increase. It was reported that the vacancies, existed during first-step aging, remain in the alloy after quenching, and facilitate cluster formation during the second step of aging [8]. Cluster-like precipitates gather in vacancies at the end of the second-step aging cycle and enhance the mechanical properties [8, 17, 18]. Stearnick and Wang [19] claimed that the clusters form in the second step of aging improve the alloy's mechanical properties as well. Therefore, it is logical to define the clusters formed in vacancies as a factor in improving the mechanical properties [12]. Besides, by increasing the aging time, the precipitates start to grow up which results in the strength to decrease. According to the experimental results in Fig.3-a, the aging time of the second step has almost no influence on the elongation. It could be concluded that when the temperature of the first step aging is 175 °C, increasing the time of the second step decreases the strength, but ductility remains constant.

As shown in Fig.3-b, after initial precipitation hardening at 190 °C, the second step aging at 65 °C leads to improvement in elongation in all cases, and just after 10 h the strength increases as well. It could be because precipitates formed in the first step of aging reach a stable state and form cluster-like precipitates during a short secondary time (10 h). Consequently, the precipitates are likely well distributed after first step aging at 190 °C and only short second precipitation hardening could increase both the strength and elongation of the material. Therefore, it could be concluded that the treatment 190 °C – 65 °C -10 h is the best cycle because of good tensile properties and short heat treatment time.

When the temperature of the initial age hardening is 205 °C the secondary aging could not

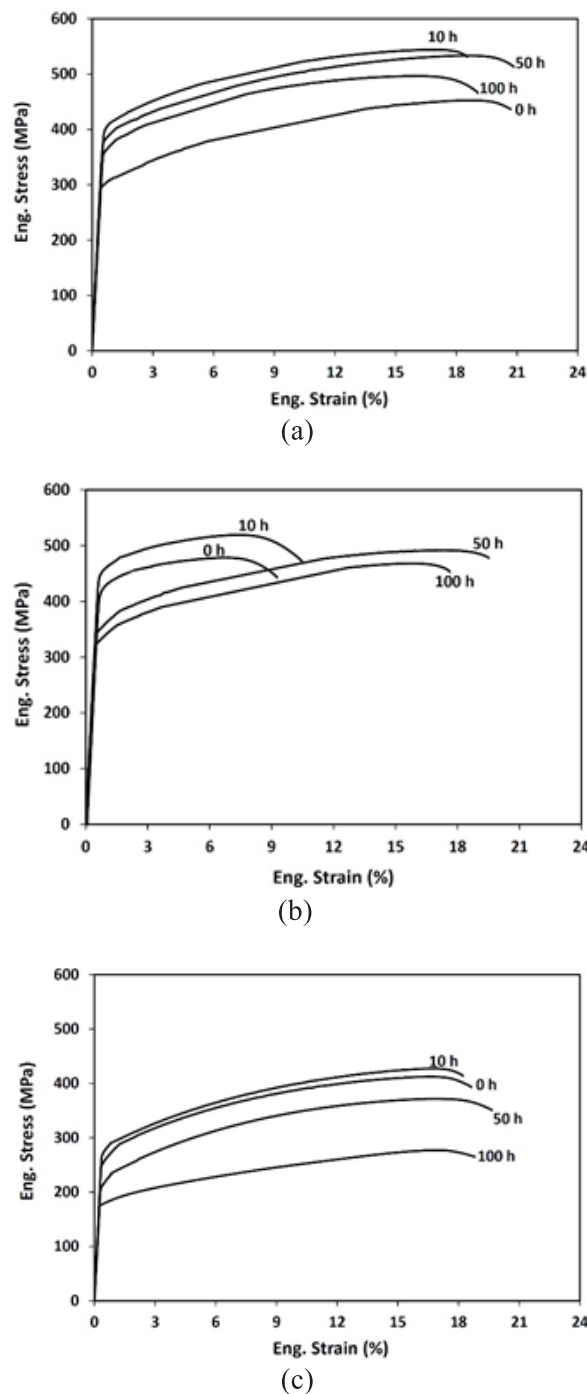


Fig. 3. Stress-strain curves after different times of the second step aging at 65 °C; first step aging at a) 175 °C, b) 190 °C and c) 205 °C.

improve the strength and ductility of the material considerably as revealed in Fig. 3-c. It could be because first aging at 205 °C results in the greatest change in microstructure. Indeed size and morphology of the precipitates which attain the stable state after the first treatment will not change

anymore by further aging. It can be concluded that in this case, the second-step aging could not get better the mechanical properties because of the over aging process occurring in the first step aging as flow curve of SA205 shows in Fig. 2.

To study the influence of temperature of first step aging and time and temperature of the second step on tensile properties of the alloy, the yield stress, tensile strength, and elongation of different heat treatment cycles in Fig.1 were compared. As shown in Fig.4, in all graphs, depending on the temperature of the first and second steps, tensile properties could be different. By increasing the second step temperature from 25 °C to 65 °C, the size, volume fraction, and morphology of precipitates change in

comparison to the ones formed in the first stage of aging. Indeed, the vacancies formed at a temperature of early precipitation hardening reach significant stability after quenching and act as nucleation sites for cluster-like precipitates at later age treatment. When the temperature of the second step aging rises from 25 °C to 65 °C, the diffusion rate increases, and kinetics of precipitation accelerate. According to the experimental results, all heat treatment conditions proposed in this study (Fig.1) except DA1752510, DA1752550, DA1902510, DA2056550, and DA20565100 lead to the yield strength, tensile strength, and elongation superior to the requirements reported in the DIN EN 755_2 standard (Table1).

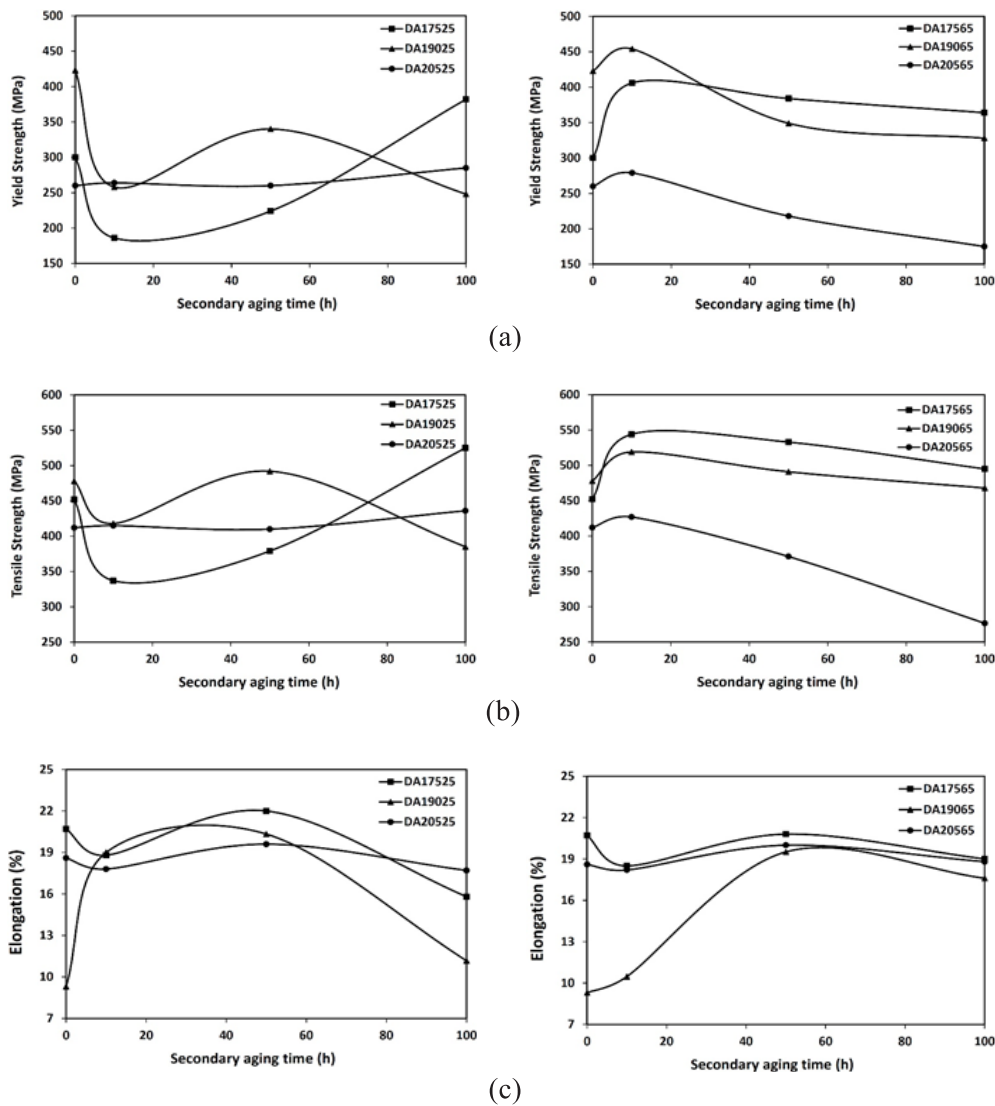


Fig. 4. Tensile properties versus secondary aging time for different temperatures of first and second step precipitation hardening; a) yield strength; b) tensile strength; c) elongation.

3.3. Hardness

Among the samples that were subjected to the single and two-step aging under different conditions, specimens with yield strength, tensile strength, and elongation greater than the value reported in the standard DIN EN755-2 (Table 1) i.e. SA190, DA17525100, DA1756510, DA1902550, DA1906510, DA20525100, and DA2056510 were selected and their hardness was measured using the Brinell hardness test method. These samples also have the maximum values between the specimens with the same heat treatment cycle. The results have been compared with the standard value which is illustrated in Fig. 5. All samples except DA20525100 and DA2056510 have a hardness greater than the value reported in the standard DIN EN755-2 which is in agreement with the results obtained from the tensile test (Fig. 4). It can be related to the more volume fraction of precipitates formed during these age treatment.

3.4. Overall Mechanical Properties and SNMP:

To determine the heat treatment cycle resulting in superior mechanical properties, the SNMP factor was calculated for different heat treatments used in this research. The factor is the sum of the normalized mechanical parameters

(which is divided by the corresponding maximum value). This parameter defined as bellow [20]:

$$SNMP = \sum \frac{\text{Mechanical Parameter}}{\text{Maximum Achieved Value}} = \frac{YS}{\text{Max. YS}} + \frac{UTS}{\text{Max. UTS}} + \frac{El}{\text{Max. El}} + \frac{HN}{\text{Max. HN}} \quad (1)$$

Among all age treatment cycles, the samples with tensile properties and a hardness greater than the value reported in the standard DIN EN755-2 in other words specimens designated by SA190, DA17525100, DA1756510, DA1756550, DA17565100, DA1902550, and DA1906510 were selected and their SNMP were determined. The results are presented in Table 4 and Fig.6. The columns correspond to each mechanical parameter that was normalized. The digit 1, bolded in each column, indicates that the parameter in that column is maximum. The last column is the sum of the normalized mechanical parameters for each aging cycle.

It can be seen in table 4 that samples undertaken two-step age-hardening including DA1906510, DA1756510, DA1756550, and DA1906510, show the maximum yield strength, ultimate tensile strength, elongation, and hardness, respectively. However, DA1756510 has generally

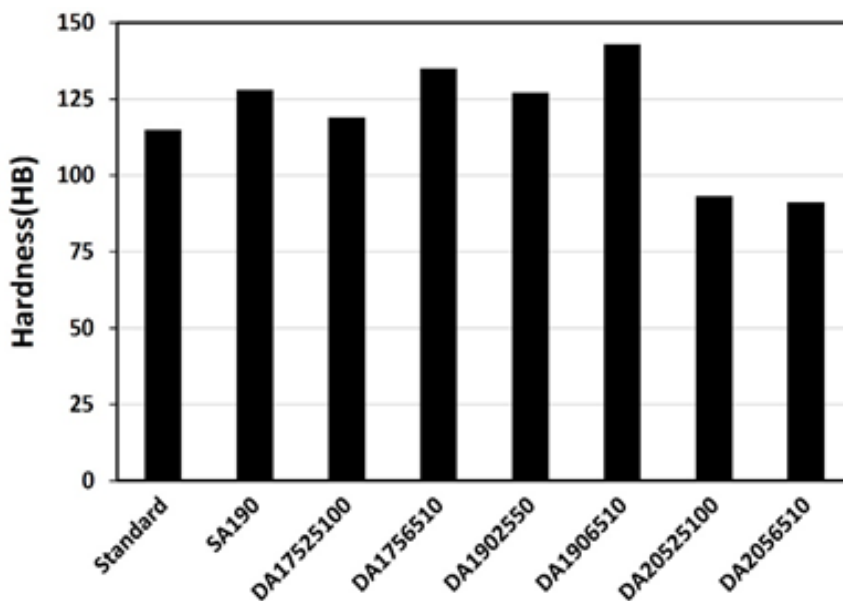


Fig. 5. Hardness values of samples owning greater tensile properties than DIN EN 755_2.

Table 4. SNMP values of heat treatment cycles with tensile properties and a hardness greater than the value reported in the standard DIN EN755-2.

| Sample code | Normalized Y. Strength | Normalized UTS | Normalized Elongation | Normalized Hardness | SNMP |
|-------------|------------------------|----------------|-----------------------|---------------------|--------------|
| SA190 | 0.932 | 0.879 | 0.447 | 0.895 | 3.153 |
| DA17525100 | 0.841 | 0.965 | 0.760 | 0.832 | 3.398 |
| DA1756510 | 0.894 | 1.000 | 0.889 | 0.944 | 3.729 |
| DA1756550 | 0.846 | 0.98 | 1.000 | 0.881 | 3.707 |
| DA17565100 | 0.802 | 0.91 | 0.913 | 0.853 | 3.478 |
| DA1902550 | 0.749 | 0.904 | 0.977 | 0.888 | 3.518 |
| DA1906510 | 1.000 | 0.954 | 0.503 | 1.000 | 3.457 |

the best mechanical properties. Therefore, the cycle, first-step aging at 175 °C, and second-step precipitation hardening at 65 °C for 10 hours is the optimum cycle. Meanwhile, the above-interrupted precipitation hardening will reduce the heat treatment time considerably compared to the conventional precipitation hardening process proposed in the literature [8, 9].

To analyze the results, the microstructure of DA1756510 having high strength and maximum SNMP was investigated by scanning electron microscopy and EDS (Fig. 7). As seen in Fig. 7-a there are some clusters and fine precipitates in the microstructure. The EDS analysis of these while secondary phases is shown in Fig7-b. The results

show that the cluster and spherical precipitates could be Mg₂ Pb and Al₂ Cu, respectively. The Mg₂Pb precipitate distributed as a cluster in the matrix. It has been reported [21-22] that the Mg₂Pb phase improves the tensile properties and hardness of Al-Cu-Mg alloy, besides the fine and distributed GP zone, θ'', θ' precipitates. The Mg₂Pb is a non-smearable precipitate because it is the non-coherent phase. But the GP zone and θ'' are both coherent and therefore they are smearable particles.

Table 5 compares the tensile properties and hardness of different aging cycles with the values of standard DIN EN755-2 in the term of normalized data. In this table, normalization

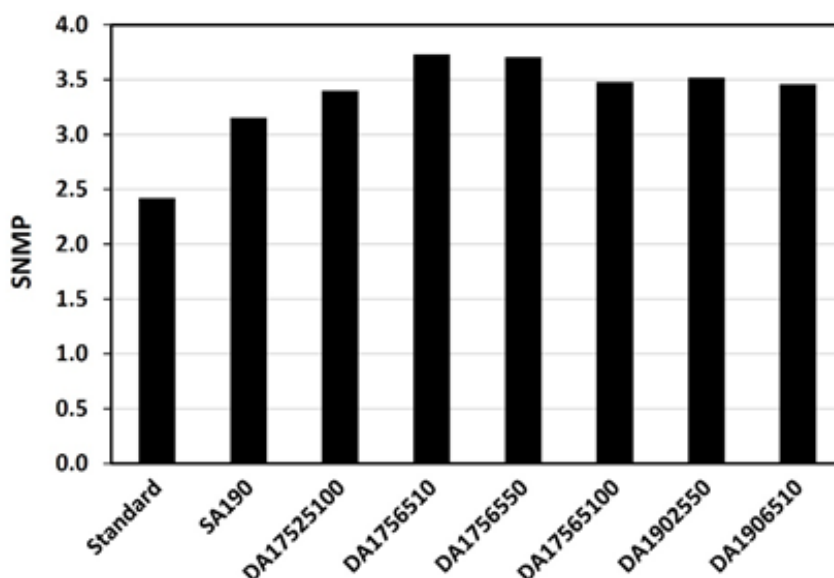


Fig. 6. Comparison of SNMP values of samples with tensile and hardness greater than the value of the standard.

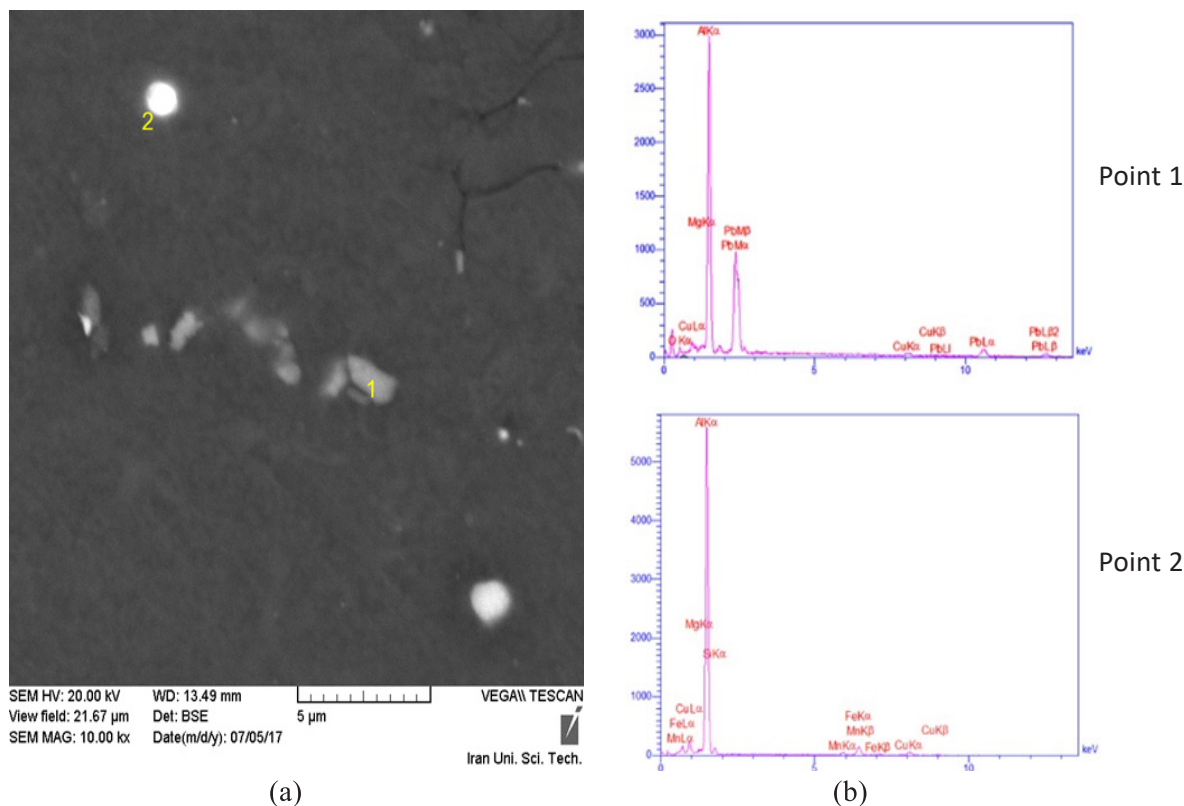


Fig. 7. a) The SEM image and b) EDS analyses of two secondary phases of DA1756510 sample.

involves dividing the yield strength, tensile strength, elongation, and hardness values into the related standard values. The results demonstrate that the mechanical properties achieved in this paper are significantly greater than the standard values. In other words, the yield and tensile strengths are about 1.5 times and the elongation is more than 2.5 times the required values.

4. CONCLUSIONS

In this work, the effect of single and double age hardening on the mechanical properties of Al-3.7Cu-1Mg alloy has been investigated experimentally to increase both strength and elongation and reduce the heat treatment time. The following results achieved:

1. The mechanical properties obtained by two-

Table. 5. Comparison between the SNMP of different age treatment cycles and standard DIN EN 755_2 values.

| Sample code | Normalized Y. Strength | Normalized UTS | Normalized Elongation | Normalized Hardness | SNMP** |
|-------------|------------------------|----------------|-----------------------|---------------------|--------|
| Standard * | 1 | 1 | 1 | 1 | 4 |
| SA190 | 1.692 | 1.292 | 1.163 | 1.113 | 5.259 |
| DA17525100 | 1.528 | 1.419 | 1.975 | 1.035 | 5.957 |
| DA1756510 | 1.624 | 1.470 | 2.3125 | 1.174 | 6.581 |
| DA1756550 | 1.536 | 1.441 | 2.600 | 1.096 | 6.672 |
| DA17565100 | 1.456 | 1.338 | 2.375 | 1.061 | 6.230 |
| DA1902550 | 1.360 | 1.330 | 2.541 | 1.104 | 6.335 |

(* from DIN EN 755_2.)

(**The summation of normalized mechanical parameters' value to the corresponding standard value.)

- step artificial aging, show improvement compared to natural ones.
- When the temperature of first-step aging rises, the second precipitation hardening decreases mechanical properties due to the formation of stable precipitates during initial age treatment.
 - Increasing the second-step artificial aging time reduces strength because of precipitation growth, but has no significant effect on elongation.
 - By introducing an SNMP factor, the interrupted precipitation hardening including first step aging at 175 °C for 2 h and second step at 65 °C for 10 h results in superior mechanical properties and because of short heat treatment time is feasible.
 - The yield and tensile strengths obtained via the proposed age treatment cycle are about 1.5 times and elongation is more than 2.5 times the values listed in standard DIN EN 755_2 for the alloy investigated in this study.

ACKNOWLEDGMENTS

The authors acknowledge financial support from The Vice-Chancellor for Research and Technology of Arak University Grant 97.3799(97.04.25).

REFERENCES

- Alexopoulos, N.D., Velonaki, Z., Stergiou, C. I. and Kourkoulis, S. K., "The Effect of Artificial Ageing heat Treatments on the Corrosion-Induced Hydrogen Embrittlement of 2024 (Al-Cu) Aluminium Alloy", *Corros. Sci.*, 2016, 102, 413–424.
- Tsai, J. M. J., "A Study of Interrupted Aging in Al-Cu-Mg Alloys", Ph.D. dissertation, The Colorado School, 2013.
- DIN EN 755-2:2013-1, <http://www.din.de/en/getting-involved/standards-committees/fnne/standards>.
- 2030 (AlCu4PbMg, A92030) Aluminum: MakeItFrom.com, <http://www.makeitfrom.com/material-properties/2030-AlCu4PbMg-A92030-Aluminum>.
- Koch, S., Abad, M. D., Renhart, S., Antrekowitsch, H. and Hosemann, P., "A High Temperature Nanoindentation Study of Al-Cu Wrought Alloy", *Mater. Sci. Eng.*, 2015, A 644, 218–224.
- Polmear, I. J., "Light alloys: from traditional alloys to nanocrystals". Elsevier, 2006.
- Löffler, H., Kovács, I., Lendvai, J., "Decomposition Processes in Al-Zn-Mg Alloys, *J. Mater.* Sci., 1983, 18, 2215–2240.
- Lumley, R. N., Buha, J., Polmear, I. J., Morton, A. J. and Crosky, A. G., "Secondary Precipitation in Aluminium Alloys and Its Role in Modern Heat Treatment, *Mater.* Sci. Forum, 2006, 519–52, 283–290.
- Lumley, R. N., Polmear, I. J. and Morton, A. J., "Interrupted Aging and Secondary Precipitation in Aluminium Alloys, *Mater.* Sci. Technol., 2003, 19, 1483–1490.
- Lumley, R. N., Polmear, I. J. and Morton, A. J., "Development of Mechanical Properties during Secondary Aging in Aluminium Alloys, *Mater.* Sci. Technol., 2005, 21, 1025–1032.
- Buha, J., Lumley, R.N., Crosky, A.G., "Secondary Ageing in an Aluminium Alloy 7050, *Mater.* Sci. Eng., 2008, A 492, 1–10.
- Marceau, R.K.W., Sha, G., Lumley, R.N. and Ringier, S.P., "Evolution of Solute Clustering in Al-Cu-Mg Alloys during Secondary Ageing, *Acta Mater.*, 2010, 58, 1795–1805.
- Risanti D., Pejrd, D.C., Bahrami, A., Miroux, A., Zwaag, S.van der, Hirsch, J., Strotzki, B., "Aluminium Alloys – their Physical and Mechanical Properties, ed". Gottstein G. Weinheim:Wiley, 2008, 1456–63.
- Risanti, D., Yin, M. Pejrd, D.C., Zwaag, S.van der, "A Systematic Study of the Effect of Interrupted Ageing Conditions on the Strength and Toughness Development of AA6061, *Mater.* Sci. Eng., 2009, A 523, 99–111.
- Gao, N., Starink, M. J., Kamp, N. and Sinclair, I., Application of Uniform Design in Optimisation of Three Stage Ageing of Al-Cu-Mg Alloys", *J. Mater. Sci.*, 2007, 42, 4398–4405.
- Cold, S., Aluminium Alloys; Aluminium - Copper 2030 Chemical Composition Mechanical Properties Standard: UNE 38323, 26–27.
- Somoza, A., Dupasquier, A., Polmear, I. J., Folegati, P. and Ferragut, R., "Positron-Annihilation Study of the Aging Kinetics of AlCu-Based Alloys", *Phys. Rev.*, 2000, B, 61, 14454–14463.
- Deschamps, A., Bastow, T. J. Geuser, F., Hill, A. J. and Hutchinson, C. R., "In Situ Evaluation of the Microstructure Evolution during Rapid Hardening of an Al-2.5Cu-1.5Mg (wt.%) Alloy", *Acta Mater.*, 2011, 59, 2918–2927.
- Starink M. J. and Wang, S. C., "The Thermodynamics of and Strengthening due to Co-Clusters: General Theory and Application to the Case of Al-

- Cu–Mg Alloys”, *Acta Mater.*, 2009, 2376–2389.
20. Mirzakhani, B. and Payandeh, Y., “Combination of Sever Plastic Deformation and Precipitation Hardening Processes Affecting the Mechanical Properties in Al-Mg-Si Alloy”, *Materials and Design*, 2015, 127–133.
 21. Lucio, L. F. and Mondolfo, F., “Aluminum Alloys: Structure and Properties”, London ; Boston: Butterworths, 1976.
 22. Seyedrezai, H., Grebennikov, D., Mascher, P. and Zurob, H. S., “Study of the Early Stages of Clustering in Al–Mg–Si Alloys using the Electrical Resistivity Measurements, *Mater*”. *Sci. Eng.*, 2009, A (525), 186–191.