INFLUENCE OF STEP-AGING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-Zn-Mg TERNARY ALLOYS

M. N. RIZWAN[a,d], A. R. MAKHDOOM[b,*], H. FAROOQ[c], M. A. KALYAR[d], I. M. GHAURI[a]
[a]*Department of Physics, GC University Lahore, Lahore 54000, Pakistan.*
[b]*Department of Natural Sciences and Humanities, Rachna College of Engineering and Technology, UET, Lahore.*
[c]*Department of Electrical Engineering, Rachna College of Engineering and Technology, UET, Lahore.*
[d]*Department of Physics, University of Sargodha, Sargodha 40100, Pakistan.*

Al-Zn-Mg alloys were prepared using RF induction furnace. Two,out of 10 samples were left un-annealed and remaining were given step-aging in temperature range 125-180°C for 2-4 hours. Characterizations of thesamples were carried out by using optical microscopy, creep and hardness tests. The results of optical micrographs reveal an equiaxed grain structure formed in all step-aged specimens that becomes more obvious with increase of aging time and temperature. The creep deformation rate was found to be minimum in two step aged specimens, whereas, hardness of the two-step aged samples is maximum as compared to the others samples.

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1. Introduction

Aluminum alloys have a significant importance and wide range of applications in manufacturing light weight structures, aerospace and automotive industry due to their better strength to density ratio, enhanced mechanical properties and easy in production [1-3]. The advantage of use of aluminum alloys is two-fold: first aluminum alloys offer low-weight and high strength. Secondly,it increases the efficiency of automobiles and reduces the energy and cost needed for transportation [4-5]. Aluminum-Zinc-Magnesium alloy is considered as one of the strongest aluminum alloys and as a result of suitable combinations and appropriate heat treatment,their strength may exceed that of steel [6-8] that make them top class candidate where better strength to density ratio is of utmost importance.

The Al-Zn-Mg ternary alloys have been the focus of many investigations and got all time considerable attention in several areas of research since the discovery of age hardening [9-17]. Stiller *et al.* [18] investigated development of precipitation in an industrial Al-Zn-Mg alloy by two step ageing treatment at 100 °C and 150 °C. Tomo Ogura *et al.*[19] explored the effects of addition of micro-alloying elements Sn and (Ag-Sn) and the two-step aging on the microstructure and mechanical properties of the Al-Zn-Mg ternary alloy. They inferred that addition of (Ag-Sn) and two-step aging process are quite effective to improve tensile properties of Al-Zn-Mg alloys. Hmon Aye *et al.*[20] carried the studies on Al-Zn-Mg alloy and investigated how to improve strength level for fuselage structure. They reported that aluminum-zinc-magnesium alloy can get the highest strength level in natural ageing.Straumal*et al.*[21] prepared high purity ternary Al-Zn-Mg alloys by vacuum induction melting and measured the heat effect of grain boundary wetting phase transition.Mahmoud Chemingui*et al.*[22] discussed the precipitation in industrial Al-Mg-Zn alloy at various steps of conventional two step ageing treatment and explored the effect of microstructure on the mechanical properties. Mohammad Tajally*et al.*[23]reported the

[*] Corresponding author: a.r.makhdoom@uet.edu.pk

comparative analysis of effects of cold working (CW) and annealing on tensile and impact-toughness behavior of Al 7075 alloy. Sharma *et al.*[24] compared the hardness strength and wear resistance values of Al-Zn-Mg alloys with magnesium free Al-Zn alloys. They optimized the concentration of zinc and magnesium which showed the best properties like strength, hardness, wear resistance and ductility. Recently, Ragab*et al.*[25]probed the effects of heating parameters on the hardness, electrical conductivity and the microstructure of Al-Zn-Mg alloys. They reported that the electrical conductivity of the alloy increases continuously with increasing aging temperature and time. While the hardness increases up to maximum value at particular aging time and temperature and decreases with further increase in aging time and temperature.

These reports show that the microstructures and mechanical properties of these alloys are strongly affected by heat treatment cycles, aging time and temperature. The motivation behind the present studies was to work out correlation in the mechanical properties and micro structure of the Al-Zn-Mg alloy as a result of heat treatment.

2. Experimental details

The Aluminum Zinc Magnesium ternary alloys were fabricated using RF induction furnace. Aluminum, Zinc and Magnesium in powder form were mixed thoroughly in the ratio 92%, 5.5% and 2.5% by weight respectively to form a homogeneous composition of alloys. The mixed powders were melted at 700 ^oC to form alloys and poured in the molds kept at room temperature. The prepared samples were heat treated at 400 ^oC for one hour and then allowed to cool them at room temperature**.** The samples were then aged at 125-180 ^oC in steps for different aging times. Creep studies and Vicker's hardness test were carried out for the study of mechanical properties. Optical microscopy was used to study the co-relation of microstructures and mechanical properties of alloys.

The samples were polished form coarse to fine grade by using silicon-carbide papers (220-4000) with the help of struer's Knuth-Rotor-3. Water was used to rinse and to flush away the removed surface products. The intermediate polishing was followed by fine polishing using diamond paste of 1μm particle size. The samples were heated at 400^{o}C for one hour in a muffle furnace and allowed to cool at room temperature. The samples were then aged at 125-180 ^oC in steps for different aging times as given in Table 1 and were allowed to cool at room temperature. The prepared samples of aluminum-zinc-magnesium alloys were used for mechanical testing and micro structural analysis. Two sets of samples with different dimensions, one having dimension $6 \times 6 \times 3$ mm^3 and other having $25 \times 6 \times 3$ mm^3 are prepared simultaneously under same conditions as given in Table 1. The grain size, shape and distribution of various phases which greatly affect the mechanical properties of the material were investigated with microscopic study of specimen. Metallic Specimens to be examined through microscopy were polished to be flat, scratch free mirror like surface and then chemically etched. The creep test on each sample of Al-Zn-Mg alloy was carried out at a constant stress of 300 Mpa and temperature of 200 ^oC. The hardness of the material is calculated by dividing the applied force to the contact area in the surface by the indentation. The indentations were made with the help of a vicker's hardness tester.

Table 1. Heat treatment cycle adopted for different samples.

Sr. No.	Sample	Aging time
1	S_1	un-annealed
2	S_2	8 hr at 180^{o}C
3	S_3	4 hr at 125^{o}C + 4 hr at 180^{o}C
4	S_4	3 hr at 125^{o}C + 2 hr at 220^{o}C + 3 hr at 180^{o}C
5	S_5	3 hr at 125^{o}C + 2 hr at 150^{o}C + 3 hr at 180^{o}C

3. Results and discussion

The microstructure of Al-Zn-Mg alloys is examined by optical microscope and the resulting structures of the un-annealed and annealed sample are shown in Fig. 1 (a, b, c, d, & e). The microstructure formed in the un-annealed alloy (sample S_1) and the single step aged alloy at 180 ^oC for 8 hours (sample S_2) shows two kinds of precipitates, the dark gray $Al_2Mg_3Zn_3$ and the light gray $MgZn_2$[17, 23, 26, 27]. The dark gray precipitates are mainly accumulated at the grain boundaries, whereas the light gray precipitates are located within the grains [28]. Fig. 1(a) shows that the microstructure of un-annealed sample, S_1, consists of equiaxed grains with well-defined and sharp grain boundaries however; for the single step-aged specimen S_2, both the precipitates are inhomogeneously distributed throughout the surface as shown in Fig. 1(b).

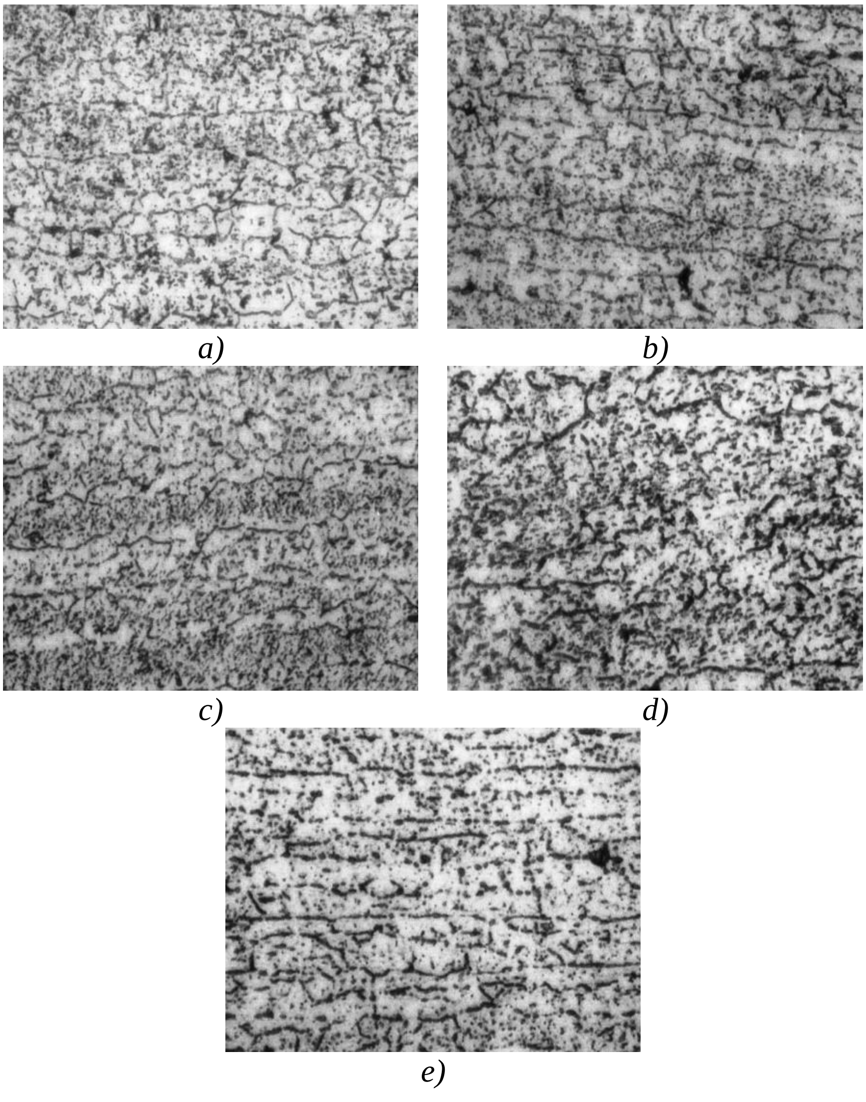

Fig.1. The microstructure (magnification 200×) of Al-Zn-Mg alloys captured by optical microscope (a) un-annealed, S_1 (b) single step aged, S_2,(c) two steps aged, S_3, (d) three steps aged, S_4 and (e) three steps aged, S_5.

Moreover, their density is low and inter spacing distance is larger as compared to those precipitates observed in the case of un-annealed specimen. The microstructure of two step-aged specimen, S_3, shows the equiaxed grain structure having both dark and gray precipitates as shown in Fig. 1(c) which are distributed uniformly within the grains and at the grain boundaries. Fig. 1(d-e) show the microstructure of the three-step aged Al-Zn-Mg alloy S_4 and S_5, respectively. The micrograph image of S_4 shows the formation of both dark and gray precipitates which are non-uniformly distributed within the grains and at the grain boundaries. This non uniform distribution of precipitates occurs due to the increase of dissolution of precipitates at high temperature (220^{o}C); the density of precipitates formed during the primary aging also decreases. The major features of this micro structure is that the coarsen grain boundary precipitates are separated from each other; they are not interconnected as in case of two step-aged samples. The equi-axed grains with uniform distribution are observed in three-step-aged specimen, S_5. Because of large atomic size, magnesium has tendency to segregate on the grain boundaries of Al-Zn-Mg alloys microstructures. The precipitates with magnesium rich contents segregate themselves at the grain boundaries thus causing the coarsening of the grain boundaries as compared to the other samples.

The changes in the grain size of the specimens have been found anomalous with aging. The average grain size of un-aged specimen as calculated using the line intercept method is found to be 77 µm. However, in the case of single step-aged specimen the grain size decreases to 68 µm. With two-steps aging of the specimen, the grain size reduces to 49 µm. The grain size is increased again to 87 µm when the three-steps aging of the specimen S_4 was carried out at 125^{o}C for 3hrs + 2hrs at 220^{o}C + 3hrs at 180^{o}C. With the change of three-steps aging temperature and time for sample S_5 (at 125^{o}C for 3hrs + at 150^{o}C for 2hrs + at 180^{o}C for 4hrs), the grain size decreases to a value of 62 µm.

Fig. 2 shows a comparison of creep curves of un-annealed sample S_1, single step-aged specimen S_2 and the two-steps aged specimen S_3 each deformed at 200 ^{o}C and at a stress level of 300 MPa. The curves show mainly two regions; in primary region, the creep deformation occurs instantaneously with time while in the secondary region the steady state creep deformation takes place. The comparison of the curves reveals that the steady state creep rate of the single step aged specimen, S_2, is higher than that of the un-aged specimen, S_1. However, interestingly the steady state creep rate of the two step-aged specimen, S_3, was found to be significantly lower than that of the single step aged specimen S_2 and un-aged specimen S_1.

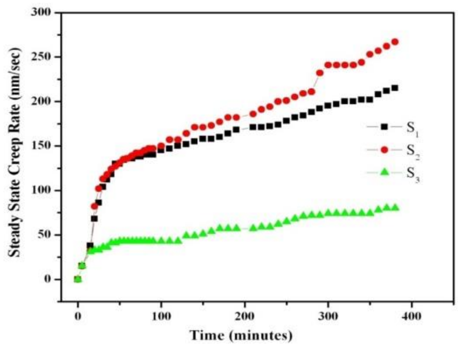

Fig. 2. A comparison of creep curves of un-annealed sample, S_1, single-step aged specimen, S_2 and the two-step aged specimen, S_3, each deformed at 200^{o}C and a stress level of 300MPa.

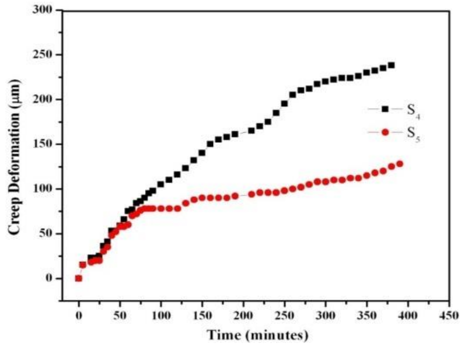

Fig. 3. A comparison of the creep curves of three-step aged specimens, S_4 and S_5, each deformed at 200^{o}C and a stress level of 300 MPa.

The values of the steady state creep rate for the un-aged, single step aged and the two steps aged specimens were found to be 4.19 nm/sec, 6.77 nm/sec and 1.88 nm/sec respectively. Similarly, the comparison of the creep curves of three steps aged specimens at various temperatures and times is shown in Fig. 3 under the same deformation temperature and stress that are used for specimens S_1, S_2 and S_3. From Fig. 3, it can be observed that steady state creep rate increases sharply in S_4 and is significantly higher than that of S_5.The creep deformation is also found to be inhomogeneous in both types of specimens. The values of the steady state creep rate were found to be 9.30 nm/sec and 3.22 nm/sec for the two specimens S_4 and S_5 respectively. The decrease in steady state creep deformation rate is attributed to the fine dispersion of the precipitates within the grains and at the grain boundaries. Whereas, the increase in steady state creep deformation rate is ascribed to the coarsening of the precipitates in the grain boundaries thus causing a decrease in their density and increases in their spacing. The results of steady state creep rate and hardness tests are listed in Table 2.

Table 2.Vicker's hardness and steady state creep rate for different samples.

Sr. No.	Sample	Steady state creep rate (nm/sec)	Micro hardness (VHN)
1	S1	4.19	73.5
2	S2	6.77	74.2
3	S3	1.88	76.1
4	S4	9.30	73.3
5	S5	3.22	75.5

Fig. 4 shows the variation in hardness and the steady state creep rate of un-aged and aged samples. The hardness of two steps aged specimen, S_3, is found to be slightly higher than that of single step aged,S_2, and the un-aged specimen, S_1. With three steps aged specimen, S_4, the hardness is found to be decreased, however, the three steps aged specimen, S_5,hardness is increased again. The two-step aged specimen shows fine dispersion of precipitates, whose density is larger than that of un-annealed and single step aged specimen as indicated in Fig. 1. The fine scale precipitation is produced in alloy when it is given a prolonged exposure at slightly elevated temperature and this primary precipitation provides basis for the formation of secondary precipitation at high temperature and leads to increase in hardness of an alloy [29]. It is evident from Fig. 4 that for the two-step aged sample, hardness is maximum with minimum creep rate. As the creep rate increases in case of three-step aged sample, S_4, the hardness of the sample reduces.

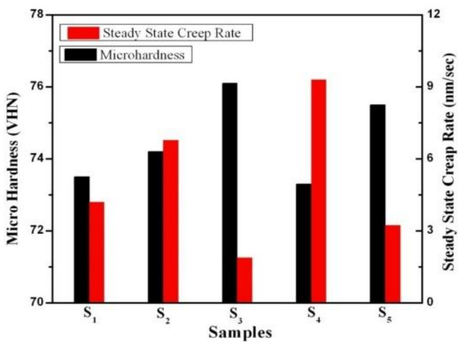

Fig. 4. Variation of Vicker's Micro hardness and steady state creep rate versus un-aged and step-aged samples.

4. Conclusions

The effects of step aging on the creep and hardness of indigenously fabricated Al-Zn-Mg alloys are investigated. The results obtained in this work have been correlated with changes in microstructures of the specimens. The microstructure studies revealed both the dark gray $Al_2Mg_3Zn_3$ and the light gray $MgZn_2$precipitates and the step aging of the samples produces the equiaxed grain structure with well-defined and sharp grain boundaries. The step aging of the specimens produced anomalous changes in their steady state creep rate. Initially with the single step aging, the creep rate is increased, however, with two steps aging it is found to be decreases. In case of three steps aging the creep rate is found to be increased further as compared to two steps aging. The changes in the micro-hardness of the alloys are found to be random with the aging steps. The anomalous changes in the steady state creep rate and micro-hardness are attributed to the non-uniform distribution of precipitates formed as a result of steps aging of the specimens.

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