Research Article

The Influence of Nickel and Tin Additives on the Microstructural and Mechanical Properties of Al-Zn-Mg-Cu Alloys

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Received 16 October 2013; Accepted 3 January 2014; Published 19 February 2014

Academic Editor: Pavel Lejcek

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The effects of nickel and nickel combined tin additions on mechanical properties and microstructural evolutions of aluminum-zinc-magnesium-copper alloys were investigated. Aluminum alloys containing Ni and Sn additives were homogenized at different temperatures conditions and then aged at 120°C for 24 h (T6) and retrogressed at 180°C for 30 min and then reaged at 120°C for 24 h (RRA). Comparison of the ultimate tensile strength (UTS) of as-quenched Al-Zn-Mg-Cu-Ni and Al-Zn-Mg-Cu-Ni-Sn alloys with that of similar alloys which underwent aging treatment at T6 temper showed that gains in tensile strengths by 385 MPa and 370 MPa were attained, respectively. These improvements are attributed to the precipitation hardening effects of the alloying element within the base alloy and the formation of nickel/tin-rich dispersoid compounds. These intermetallic compounds retard the grain growth, lead to grain refinement, and result in further strengthening effects. The outcomes of the retrogression and reaging processes which were carried on aluminum alloys indicate that the mechanical strength and Vickers hardness have been enhanced much better than under the aging at T6 temper.

1. Introduction

With the growing demands of the airline industries, various efforts were taken to develop modern advanced structural materials. Super high-strength aluminum alloys (Al-Zn-Mg-Cu) have attracted much attention in the aerospace fields due to their excellent combination of low density and high-strength [1, 2]. Recently, attempts, including a chemical composition modification for an Al-Zn-Mg-Cu alloy and employing new heat treatments, were made to further increase the properties of these alloys. Zhao and Tsuchida [3] found that adding chromium (Cr) or zirconium (Zr) into AA 7075 aluminum alloy could inhibit the grain coarsening. Chaubey et al. [4] observed that an addition of cerium (Ce) into Al-Zn-Mg-Cu alloy has resulted in up to 5% grain refinement of the cast dendritic structure as well as up to 38% refinement of heat-treated microstructure. However, the applications of the rare-earth metals contained in aluminum alloys are extremely

restricted due to their high cost. Therefore, more attention has been paid to the transition metals which are cheap such as nickel. Previous research showed that nickel plays an important role in solid solution strengthening and can effectively improve the mechanical properties of Al-7Si alloys [5]. Farkoosh et al. [6] assessed the phase formation in an Al-Si-Cu-Mg-Ni alloy through adding nickel (0-1 wt.%); also, the Al₃CuNi phase has greater influence on the overall strength of the alloy compared to other Ni-bearing precipitates. At present, studies on Al-Zn-Mg-Cu alloys are developed which modified most of them by nickel with various techniques like a rapid solidification (RS) [7, 8]. While the researchers on the impacts of nickel and tin additives which added to an Al-Zn-Mg-Cu alloy by the another casting process, especially they were a joint effect, is very few. In the present work, the influences of Ni and Sn on the microstructure and mechanical properties of Al-Zn-Mg-Cu alloys produced by a semichilling casting process were investigated. In order to optimize the

TABLE 1: The chemical composition of the alloys.

Alloy	Chemical composition (wt. %)										
	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Sn	Al
Base alloy	0.078	0.234	1.774	0.044	2.886	0.187	—	6.676	0.027	—	Bal.
Alloy A	0.092	0.220	1.606	0.044	2.756	0.185	0.557	6.683	0.043	—	Bal.
Alloy B	0.130	0.389	1.641	0.46	2.817	0.181	0.57	7.615	0.048	0.511	Bal.

TABLE 2: The heat treatment steps of studied alloys after the extrusion process.

Number of treatment	Type	Details of treatment
1	Homogenizing	450°C for 2 h + 470°C for 24 h + 480°C for 0.5 h
2	Peak ageing (T6)	120°C for 24 h (peak ageing)
3	Retrogression and reaging (RRA)	120°C for 24 h (peak ageing) + 180°C for 0.5 h (retrogression) + 120°C for 24 h (reaging)

role of Ni and Sn additives on Al-Zn-Mg-Cu alloys, the retrogression and reaging were carried out on these alloys.

2. Experimental Procedures

2.1. Research Material. The present study was carried out on Al-Zn-Mg-Cu aluminium alloy ingots provided by ALCAN GLOBAL AEROSPACE. The ingots were 13 mm thick and 20 mm wide. Nickel and tin granular additives of 99% and 99.5% purity, respectively, were provided by Merck KGaA. The nominal compositions of the studied alloys are listed in Table 1. The terms “Base alloy,” “Alloy A,” and “Alloy B” refer to the as-received alloy, alloy with 0.5 wt.% Ni, and alloy with a combined 0.5 wt.% Ni plus 0.5 wt.% Sn additions, respectively. The chemical composition analysis was carried out using the arc-spark spectrometer.

The alloys were remelted in a graphite crucible at 1123 K in an electrical resistance furnace (with accuracy of $\pm 5^\circ\text{C}$). The samples were produced by a semidirect chilling (DC) casting process conducted in a cylindrical iron steel mold of dia._{in-out} 35–45 mm² × height 150 mm. The mold was preheated to 523 K prior to the casting process. The cooling rate of -7 K/s and water flow rate were about 45 liters per minute. The alloys were inverted and remelted three times to ensure complete mixing. After the casting, homogenizing treatments conducted for alloys according to [9, 10], within the step number 1 in Table 2, were followed by quenching in cool water immediately after each step of the homogenizing treatments. After quenching, an extrusion process was performed on the cast bars. In order to accomplish so, the alloy bars and extrusion mold were preheated to 400°C and to 450°C for 30 minutes in electrical resistance furnaces. After extrusion, the samples were quenched in cool water. The extrusion rate was about 3.5. During the extrusion process, wax was used as a lubricant material. The extruded bars were undergoing separately at 450°C and 470°C for 1 h and then kept at 480°C for 30 min for further homogenizing treatment. Finally, they were quenched in cold water immediately after each step. In this study, all the extruded samples were tempered according to the procedure in Table 2. After each step of the heat

treatment T6 temper and RRA process, the specimens were quenched in cool water.

2.2. Microstructure Characterizations. The microstructures were analyzed by the optical microscopy (OM) using Olympus PMG3 optical microscope. The specimens were extracted from a position of 1/2 height of the ingot, ground, and polished according to ASTM E3-01. They were etched with Keller’s reagent. The average grains’ size analysis was carried out using the linear intercept method. To characterize the effect of additives on the microstructural, the scanning electron microscopy (SEM-JEOL JSM-6460LA analytical scanning electron microscope) along with an energy dispersive spectroscopy (EDS) was used. The X-ray diffraction analysis (XRD-Lab X, XRD-6000, SHIMADZU, under the operating conditions: scan range: 20°–80°, step size: 0.03, and scan rate: 5°/min) has been used to identify the intermetallic compounds of alloys. The calculation of the average grain size and its analysis were carried out using the linear intercept method.

2.3. Mechanical Testing. The Hv microhardness measurements were carried out on the specimens according to ASTM E92-82, “Mitutoyo DX256 series.” Indentation force was set to 30 N and 10 sec dwell time. To ensure cleanliness the surfaces of the samples were polished prior to Hv measurement. Each reading was an average of at least ten separate measurements taken randomly on the surface of the specimens.

The tensile test was carried out at an ambient temperature on a round tensile specimen with dimension of length 90 mm × dia. 65 mm² using an INSTRON testing machine with a ram speed of 10 mm/minute and a load of 500 kN. Three rods were tested to ensure the right results. The tensile test specimens were prepared according to ASTM B557M-02a.

3. Results

Figures 1(a) and 1(b) show the optical micrographs of the as-quenched alloy A and alloy B samples. The structure of samples casting consists of the equi-axed grains are clear

FIGURE 1: Optical microstructures of as-quenched (a) alloy A and (b) alloy B samples.

FIGURE 2: Optical microstructures of (a) alloy A and (b) alloy B after T6 temper.

within aluminum-rich solid solution this equiaxed due to the effectiveness of the semi-chill casting mechanism an addition to impact of the nickel additives cause of the interdendritic network of the intermetallic compounds. In general, grain refining is normally employed during chill casting mechanism; the structure refined by increasing the heat extraction occurs from the surface and corresponding increase at the solidification rate [11]. The grain size is reduced for the alloys A, B in Figures 1(a) and 1(b) with adding nickel to the base alloy. Generally, the idea behind a decrease in the grain size is attributed to the grain refinement mechanism which is the increase as the number of solidification sites for heterogeneous nucleation of the primary aluminum phase [12, 13]. Particles of a nickel act as substrates within the matrix base alloy. The grain refiner effectiveness is judged through the grain size and morphology, where if the grain morphology was equiaxed, this leads to a finer structure which in turn allows for advantages of grain refinement such as improved mechanical properties [14].

Further the average grain size of the as-quenched alloy A and alloy B samples is about 39 and 43 μm, respectively; compare with the as quenched base alloy in our prior study where the average grain size of was 47 μm [15]. Previous studies indicate an average grain size; Deng et al. [16] reported a grain size of about 121 μm for as-cast AA 7050 aluminum alloy. He et al. [17] gave the grain size about 250 μm for as-cast Al-Zn-Mg-Cu alloy. Semi-direct chill casting findings in this study confirmed the efficiency of the technique in the grain refinement and enhancement of mechanical properties.

Figures 2(a) and 2(b) show the optical micrographs of the alloy samples after promoting homogenizing and hot extrusion and then the aging treatment at T6 temper. The average grain size was about 31 and 36 μm for alloy A and alloy B samples, respectively.

Figures 3(a) and 3(b) show the optical micrographs of the alloy A and alloy B specimens after the RRA process. The average grain size of these alloys was about 33 and

FIGURE 3: Optical micrographs of (a) alloy A and (b) alloy B after the RRA process.

FIGURE 4: The (a) SEM and (b) EDS microanalysis of as-quenched alloy A sample.

39 μm , respectively. The grain size of alloy A samples in Figures 2(a) and 3(a) was significantly reduced after applying the T6 and RRA processes due to the increase to extensive hot extruding process. Furthermore, with adding nickel into aluminum alloys wherein the eutectic reaction led to creating dispersion compounds within the matrix of the alloys, the interaction of the base alloy matrix underwent heat treatment (T6, RRA) with the nickel-rich disperse particles. The newly formed compound of the dispersed particles restricts the recrystallization and grain growth in subsequent stages.

The scanning electron micrograph (SEM) in Figure 4(a) depicts the microstructure of the as-quenched alloy “A” sample. The dark areas indicate the primary solid solution, and the bright areas indicate the nonequilibrium eutectic solid solution between the grains. Gray particles in the labeled

region are prevalent; the energy-dispersive spectroscopy (EDS) scanned analysis in Figure 4(b) suggests that the stoichiometry is similar to T-(Al Mg Zn) and S-(Al Cu Mg) phase with γ -(Al-Cu-Ni-Fe) phases.

The SEM micrograph of the alloy “A” sample after T6 temper in Figure 5(a). It shows the prevalence of Ni-rich dispersoids particles within the matrix. Figure 5(b) reveals the chemical composition close to T-(Al Mg Zn) and S-(Al Cu Mg) phase with γ -(Al-Cu-Ni-Fe) phases.

SEM image in Figure 6 shows the microstructure of the alloy “A” sample undergoing RRA process. The bright areas denote the newly formed phases in addition to the dispersoids particles. Figure 6(a) shows the numerous dispersed particles. Figure 6(b) reveals a similar stoichiometry to that of T-(Al Mg Zn) and S-(Al Cu Mg) phase with γ -(Al-Cu-Ni-Fe)

FIGURE 5: The (a) SEM and (b) EDS microanalysis of alloy "A" sample after T6 temper process.

FIGURE 6: The (a) SEM and (b) EDS analysis of alloy "A" sample after the RRA process.

phases. During the RRA process further the secondary phase particles are precipitated into the matrix.

Figure 7 shows comparison of the X-ray diffraction (XRD) patterns of alloy A after quenching, T6 temper, and RRA process. The as-quenched alloy "A" XRD pattern in Figure 7(c) was primarily composed of the alpha aluminum, the secondary phases, and intermetallic compounds (T- $\text{Al}_5\text{Mg}_{11}\text{Zn}_4$, S- Al_2CuMg , $\text{Al}_7\text{Cu}_4\text{Ni}$, $\text{Al}_{50}\text{Mg}_{48}\text{Ni}_7$, Mg_2Zn , Al_4Ni_3 , $\text{Al}_{75}\text{Ni}_{10}\text{Fe}_{15}$, and Al_3Ni_2). The outcomes of the XRD analysis indicated coexistence of nickel-rich intermetallic compounds within the matrix of the alloy A. According to the phase diagrams for an Al-Ni, Al-Cu-Ni and Al-Fe-Ni phases

by [18] showed that Nickel could combine with Al, Cu, and Fe to form the intermetallics.

The generally accepted precipitation sequences for a 7000 series aluminum alloys are as follows: supersaturated solid solution $\rightarrow$ coherent stable Guinier-Preston (GP) zones $\rightarrow$ semicoherent intermediate η' ($\text{Mg}_2\text{Zn}_{11}$) phase (metastable η (MgZn_2) or T ($\text{AlMg}_4\text{Zn}_{11}$) phase) [19, 20]. The primary precipitations in the matrix are the GP zones and the η' phase after aging at 120°C for 24 h.

The XRD plots in Figure 7(b) show that the alloy "A" sample after T6 temper exhibits dispersoid compound phases ($\text{Mg}_2\text{Zn}_{11}$, MgZn_2 , $\text{Al}_{75}\text{Ni}_{10}\text{Fe}_{15}$, and Al_4Ni_3) in addition to

FIGURE 7: The XRD plots for alloy A: after quenching, T6 heat treatment, and RRA process.

the compounds already existing in the as quenched sample of alloy A. These dispersive phases have high peaks as a result of intensive dissolution of the alloying elements and the nickel additives in the matrix which are brought by homogenization, extrusion, and subsequent heat treatments. On the other hand, Li et al. [21] found that adding nickel to an Al-Zn-Mg-Cu alloy suppresses the formation in the MgZn_2 phase in the matrix. Findings of Li et al. came in contradiction to the outcomes of EDS and XRD analysis through this study which revealed plentiful of MgZn_2 phases.

The XRD analysis in Figure 7(a) for the alloy “A” sample after the RRA process indicates high-intensity diffraction peaks of the Al_4Ni_3 , Al_3Ni_2 , $\text{Al}_{75}\text{Ni}_{10}\text{Fe}_{15}$, and η (MgZn_2) with η' ($\text{Mg}_2\text{Zn}_{11}$) phases. Generally it had been suggested by Li et al. [22] that during the high temperature of the retrogression process (180°C for 30 minutes as in this study) undissolved GP zones transformed into η' phase and thus formed numerous GP zones and η' phases. The results were consistent with XRD results through this study. Finally, plenty of the GP zones and η' -phase ($\text{Mg}_2\text{Zn}_{11}$) re-precipitation during the reaging step.

The SEM micrograph in Figure 8(a) shows the microstructure of the as-quenched alloy “B” sample. The dark areas denote the primary solid solution. Gray particles are observed in the encircled region (X). The EDS microanalysis detects the reduced concentrations of magnesium and zinc noticing that the stoichiometry of tin (Sn) has the high concentration (Figure 8(b)). The bright areas denote the nonequilibrium solidification eutectic system between grains as indicated in the enlarged labeled region (Figure 8(c)).

The SEM micrograph in Figure 9(a) of the alloy B sample after T6 temper indicates the prevalence of the Ni-affluent dispersoids particles. The encircled region (Y) shows a rod shape as in Figure 9(c). The EDS microanalysis results of this region in Figure 9(b) reveal the chemical composition close to the T-(Al Mg Zn) and the S-(Al Cu Mg) phase with γ -(Al-Cu-Ni-Fe) phases and Al Cu Sn phase.

Figure 10 shows the microstructure of the alloy A sample after the RRA process. Figure 10(a) shows the numerous dispersion particle phases as bright areas and the region (Z) indicates Chinese script morphology (see Figure 10(c) for highly magnified SEM). Figure 10(b) reveals similar stoichiometry to T-(Al Mg Zn) phase and S-(Al Cu Mg) phase with γ -(Al-Cu-Ni-Fe) phases, (Al-Cu-Sn) and (Al-Ni-Sn) phases. Sn element is peaked highly in the EDS analysis.

The XRD analysis results of the alloy B samples are shown in Figures 11(a)–11(c) (RRA, T6, and as-quenched, resp.). The patterns of the as-quenched alloy B sample confirm that the principle eutectic mainly consisted of α (Al), solid solution, and intermetallic compounds.

Figure 11(b) shows the XRD plots of the alloy B sample undergoing T6 temper which indicates coexistence of the dispersoids particles of the $\text{Al}_7\text{Cu}_4\text{Ni}$ and $\text{Al}_4\text{Ni}_{15}\text{Sn}$ in addition to the compounds which were peaked highly such as Al_4Ni_3 , $\text{Al}_{75}\text{Ni}_{10}\text{Fe}_{15}$, and $\text{Al}_3\text{Cu}_{12}\text{Sn}$. Dispersed phases possessed high peaks because of the intensive dissolution of the alloying elements and the nickel combined with Sn element produced by a series of the homogenization and subsequent heat treatments.

Figure 11(a) shows the XRD plots for the alloy B sample after the RRA process. The intensity of the diffraction peaks $\text{Al}_4\text{Ni}_{15}\text{Sn}$ and other peaked high such as $\text{Al}_7\text{Cu}_4\text{Ni}$, Al_4Ni_3 , $\text{Al}_{75}\text{Ni}_{10}\text{Fe}_{15}$ and $\text{Al}_3\text{Cu}_{12}\text{Sn}$ phases with a little coexistence of the $\text{Mg}_2\text{Zn}_{11}$ phase. The reason standing behind disappearance of some of the compounds in the MgZn phase through adding 0.5 wt.% Sn into the alloy A is attributed to the impacts of tin (Sn) in the nucleation within the vicinity of the grain boundaries which is resulting in the suppression of some of the sites, the creation for the MgZn phases within the matrix alloy B in this study present. This justification conforms with prior researchers: Ogura et al. [23] observed that adding tin element into Al-Zn-Mg alloy led to precipitates suppression of the MgZn phases which are sparsely formed within the microstructure. The outcomes of XRD analysis of alloy B conform to the EDS scan results as noticed in Figure 10(b).

After applying the aging at T6 temper and RRA process for the alloy A and alloy B, the yield maximum gains of about 385 and 415 MPa and 370 and 385 MPa were attained in the ultimate tensile strength (UTS), respectively. Figure 12 shows the variation in the UTS value among samples of the as-quenched alloys and same the samples which underwent T6 temper and the RRA process. This is due to the grain refinement and the evolution of the new interdendritic networks brought about by the intensive extrusion process and the heat treatment processes as shown earlier in Figures 1 and 2.

Figure 13 shows the variations in the Vickers hardness of the alloy specimens under different heat treatments. Generally, alloys A and B are gaining about 110 and 120 HV, respectively, after undergoing the T6 and RRA process. Principally the hardness scale depends on the extrusion process which led to reducing the grain size as well as the influence of the ageing treatment which was included on the distribution of precipitates and dispersion phases of the additives in the matrix during these processes.

FIGURE 8: The (a) SEM micrograph and (b) EDS microanalysis for the as-quenched alloy B sample.

FIGURE 9: The (a) SEM micrograph and (b) EDS microanalysis of alloy B sample after having undergone T6 heat treatment.

FIGURE 10: The (a) SEM micrograph and (b) EDS microanalysis of alloy B sample after having undergone RRA process.

FIGURE 11: The XRD plots for alloy B (a) after the RRA process (b) after the T6 heat treatment and (c) the as-quenched.

4. Discussion

The results indicated that the yield strength (YS), ultimate tensile strength (UTS), and Vickers hardness of both alloys A and B substantially improved after the T6 temper and RRA treatment.

The strengthening mechanisms alloys (A and B) divided into the precipitation hardening in addition to dispersion strengthening. The precipitation hardening was detailed by [20–22]. The dispersion strengthening can be described as

dislocations inhibited by Ni or/and Sn dispersoid in the slipping planes. Assumption of dispersoids, that the dispersed phase particles were looped, bypassed, and/or sheared by dislocation through the Orowan mechanism. The stress required to move a dislocation around a particle is YS, which is increased by dispersion strengthening. So the increase in the yield strength (YS) due to the Orowan strengthening, $\Delta\sigma_y$, Or was given by [24, 25]

$$\Delta\sigma_y, Or = \frac{2Gb}{Ld}, \quad (1)$$

where G is the shear modulus of the matrix, b is Burger's vector of the dislocation, and Ld is the interparticle spacing of the dispersoids. The XRD analysis indicated that the extrusion and the heat treatments (T6 temper and the RRA process) on alloys A and B have resulted in lower Ld spacing as compared to the Ld values of the parent alloy phases as shown in Table 3. Thus according to (1) the yield strength is high for the alloys A and B.

5. Conclusions

- (1) Microstructures observations for alloys revealed the presence of phases such as Al₇Cu₄Ni, Al₄Ni₃, Al₅₀Mg₄₈Ni₇, Al₇₅Ni₁₀Fe₁₅, Al₃Ni₂, Al₄Ni₁₅Sn, and Al₃Cu₁₂Sn which are standing behind the dispersoids particles through heat combined with mechanical treatments.

FIGURE 12: Variations of the tensile strength for alloys samples after different heat treatments.

FIGURE 13: Indicating the variations in the Vickers hardness of the alloy specimens under different conditions.

TABLE 3: The XRD results of the compounds phase which appeared in the two tested alloys.

Alloys	Intermetallics compounds	<i>d</i> -spacing (Å)
Base alloy	Al ₃ Mg ₁₁ Zn ₄	2.35605
	Al ₂ CuMg	2.31249
	Mg ₂ Zn ₁₁	2.07419
	MgZn ₂	2.16920
Alloy A	Al ₇ Cu ₄ Ni	2.04391
	Al ₄ Ni ₃	2.01674
	Al ₇₅ Ni ₁₀ Fe ₁₅	2.05108
	Al ₃ Ni ₂	2.00637
Alloy B	Al ₃ Cu ₁₂ Sn	2.1010
	Al ₄ Ni ₁₅ Sn	2.0789

- (2) The mechanical properties of (Al-Zn-Mg-Cu-Ni) alloy A after the retrogression and reaging processes led to raising the ultimate tensile strength to high level to be about 630 MPa and Vickers hardness being about 250 HV.

- (3) With adequate additions of tin into (Al-Zn-Mg-Cu-Ni) alloy B, the alloy exhibited the strength and hardness values: yield strength 534 MPa, ultimate tensile strength about 595 MPa, and Vickers hardness 240 MPa were which attained after the RRA treatment. The strength of alloy B was lower than alloy A because of adding tin element which suppresses some of the MgZn compounds phases.
- (4) Generally, the incremental increase in the strength of alloys A and B through the present study was due to the precipitation hardening and Orowan strengthening which are working together.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

This work is supported under Grant no. 9001-00338 of the University Malaysia Perlis (UniMAP). The authors gratefully

acknowledge the outstanding support provided by the technicians of the workshop in the Materials Engineering School, UniMAP.

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