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# The Phase Composition and Mechanical Properties of the Novel Precipitation-Strengthening Al-Cu-Er-Mn-Zr Alloy

Sayed Amer<sup>1,2</sup>, Olga Yakovtseva<sup>1</sup>, Irina Loginova<sup>1</sup>, Svetlana Medvedeva<sup>1</sup>, Alexey Prosviryakov<sup>1</sup>, Andrey Bazlov<sup>1,3</sup>, Ruslan Barkov<sup>1</sup> and Andrey Pozdniakov<sup>1,\*</sup>

- <sup>1</sup> Department of Physical Metallurgy of Non-Ferrous Metals, National University of Science and Technology MISiS, 119049 Moscow, Russian; e.khamees@misis.ru (S.A.); yakovtseva.oa@misis.ru (O.Y.); loginova@misis.ru (I.L.); medvedeva71@list.ru (S.M.); pro.alex@mail.ru (A.P.); bazlov@misis.ru (A.B.); barkov@misis.ru (R.B.)
- <sup>2</sup> Faculty of Engineering, Metallurgy and Petroleum Engineering Department, Al-Azhar University, Mining, Cairo 11371, Egypt
- <sup>3</sup> 7/9 Universitetskaya nab, St.Petersburg State University, 199034 St. Petersburg, Russia
- \* Correspondence: pozdniakov@misis.ru; Tel.: +7-(495)-638-44-80

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**Abstract:** The microstructure, phase composition, and mechanical properties during heat treatment and rolling of the novel Al-5.0Cu-3.2Er-0.9Mn-0.3Zr alloy were evaluated. A new quaternary (Al,Cu,Mn,Er) phase with possible composition  $Al_{25}Cu_4Mn_2Er$  was found in the as-cast alloy.  $Al_{20}Cu_2Mn_3$  and  $Al_3(Zr,Er)$  phases were nucleated during homogenization, and  $\theta''(Al_2Cu)$  precipitates were nucleated during aging. The metastable disc shaped  $\theta''(Al_2Cu)$  precipitates with a thickness of 5 nm and diameter of 100–200 nm were nucleated mostly on the  $Al_3(Zr,Er)$  phase precipitates with a diameter of 35 nm. The hardness Vickers (HV) peak was found after the annealing of a rolled alloy at 150 °C due to strengthening by  $\theta''(A_2Cu)$  precipitates, which have a larger effect in materials hardness than do the softening processes. The novel Al-Cu-Er-Mn-Zr alloy has a yield strength (YS) of 320–332 MPa, an ultimate tensile strength (UTS) of 360–370 MPa, and an El. of 3.2–4.0% in the annealed alloy after rolling condition.

**Keywords:** aluminium alloys; erbium; phase composition; precipitates; recrystallization; mechanical properties

## 1. Introduction

Erbium is a prospective alloying element to improve the mechanical properties of pure aluminium [1–10] and Al-Mg alloys [11–17] due to the nucleation of L1<sub>2</sub> precipitates after the annealing of as-cast ingots. Zhang et al. demonstrated strengthening in the Al-0.045 at.%Er, which was achieved by Al<sub>3</sub>Er precipitate nucleation [1]. The precipitation hardening effect increases in the zirconium doped Al-Er alloys due to Al<sub>3</sub>(Er,Zr) precipitate particles [2–4]. Booth-Morrison et al. indicated that two-stage annealing provides maximum hardness because of the formation of the optimum microstructure with Al<sub>3</sub>(Er,Zr,Sc) dispersoids [5]. Erbium improves the thermal stability of the Al<sub>3</sub>(Sc,Zr) phase precipitates through annealing processes [6]. Standard silicon impurities in the aluminium have a positive effect in the strengthening effect of the Al–Zr–Sc–Er alloys due to the enhancement of the precipitate density [7,8]. Silicon in combination with Fe impurities decrease hardening due to (AlSiFeEr(Zr)) and (AlSiErY(Zr)) phase formation during solidification of the Al-Y-Er-Zr-Sc alloy [10]. The same strengthening mechanism provides good mechanical properties in the Al-Mg alloys [11–17], and Er has a positive effect in primary aluminium grain refining [12,14,15,17]. Most researchers have investigated the effect of small additives



of Er in the aluminium alloys. It was also found that quasi-binary alloys in the ternary Al-Cu-Er system may be used to develop novel cast and wrought alloys [18,19]. Ternary alloys with a 4/1 atomic rotation of Cu/Er from the quasi-binary section Al-Al<sub>8</sub>Cu<sub>4</sub>Er have a narrow solidification range (less than 40 °C) [18,19] compared with that of the Al-Cu alloys (about 100 °C) [20–23], which can provide the same casting properties found in Al-Si-Cu alloys [20–25]. Investigations of the quasi-binary alloys of the Al-Cu-Y and Al-Cu-Ce systems demonstrated low hot cracking susceptibility [26–28]. Moreover, the fine eutectic Al<sub>8</sub>Cu<sub>4</sub>Er phase demonstrates good size stability during high temperature homogenization treatment [18,19]. Zirconium addition in the ternary Al-Cu-Y and Al-Cu-Er alloys significantly improves the yield strength of the rolled alloys due to Al<sub>3</sub>(Y,Zr) [29] and Al<sub>3</sub>(Er,Zr) [30] precipitate nucleation after homogenization treatment. Additional alloying of the novel Al-Cu-Er alloys should provide an increase in mechanical properties. For example, the classical alloying element Mn in the Al-Cu alloy improves yield strength due to  $Al_{20}Cu_2Mn_3$  phase formation during homogenization treatment [22]. The present research aimed to investigate the microstructure, phase composition, strengthening mechanisms, and mechanical properties of the novel Al-Cu-Er-Mn-Zr alloy.

## 2. Materials and Methods

Pure Al (99.99%), Al-51.7Cu, Al-3.5Zr, Al-10Er, and Al-10Mn master alloys were used as an initial material to melt the alloy. Approximately 2 kg of the alloy was prepared in the resistance furnace at 750 °C and poured into a water-cooled copper mould with a final ingot size of 40 mm in width, 20 mm in thickness, and 120 mm in height. The nominal and experimental chemical composition of the investigated alloy is presented in Table 1. A Labsys Setaram differential scanning calorimeter (DSC) was used to determine the solidus temperature of the alloy.

Element	Al	Cu	Er	Mn	Zr
nominal	bal.	5.0	3.4	0.8	0.3
experimental	bal.	5.0	3.2	0.9	0.3

Table 1. Chemical composition of the alloy, wt %.

A Neophot-30 light microscope (LM), a TESCAN VEGA 3LMH scanning electron microscope (SEM) operating at 20 kV, a Bruker D8 Advance diffractometer, and a JEM 2100 transmission electron microscope (TEM) operating at 200 kV were used to evaluate the grain structure, phase composition, and precipitation types. The ingots were rolled to 1 mm thick sheets after homogenization treatment. Hardness Vickers equipment with a 5 kg load and a Zwick/Roell Z250 testing machine were used to evaluate the mechanical properties during heat treatment of the as-cast and as-rolled alloy.

## 3. Results

The aluminium phase, the Al<sub>8</sub>Cu<sub>4</sub>Er phase in the fine eutectic, and the Al<sub>3</sub>Er phase were identified by SEM and X-ray diffraction analyses (Figure 1). During solidification, 1.6%Cu, 0.9%Mn, 0.3%Zr, and 0.2–0.3%Er were dissolved in the (Al) solid solution according to the point electron diffraction X-ray (EDX) analysis in SEM. As shown by the map element distribution (Figure 1a), the Cu-, Er-, and Mn-rich phases are presented in the as-cast microstructure. Unidentified peaks (marked with "?" in Figure 1b) were found in the XRD patterns. These peaks were not found in the XRD patterns of the alloy with the same composition without Mn addition (grey line in Figure 1b). These peaks probably correspond to the quaternary (Al,Cu,Mn,Er) phase. The atomic rotation of Cu/Mn/Er in this phase is 4/2/1 in accordance with point EDX SEM results. The possible compound Al<sub>25</sub>Cu<sub>4</sub>Mn<sub>2</sub>Er may match the quaternary (Al,Cu,Mn,Er) phase. The same morphology has an Al<sub>15</sub>(Fe,Mn)<sub>3</sub>Si<sub>2</sub> eutectic phase [22,24].



**Figure 1.** (a) As-cast microstructure (SEM), (b) XRD patterns of the investigated alloy (black line) in comparison with the Mn-free alloy (grey line), and (c) DSC curves.

A homogenization temperature of 605 °C was chosen to be in accordance with the solidus temperature of 614 °C (results of DSC analysis (Figure 1c)) of the investigated alloy. Figure 2 illustrates the evaluation of the phase size, morphology, and concentration of copper in the aluminium solid solution. The Al<sub>8</sub>Cu<sub>4</sub>Er eutectic phase fragmentized and grew from 1.8  $\mu$ m after 1 h of annealing to 2.6  $\mu$ m after 3 h of annealing (Figure 2). The quaternary (Al,Cu,Mn,Er) phase did not change in size or morphology. The maximum copper content of 2.2% in the aluminium solid solution was achieved after 3 h of annealing due to the dissolving of the non-equilibrium part of the copper content intermetallic phases. The investigated alloy was quenched after 3 h of homogenization treatment and aged at 150, 180, and 210 °C. The hardness Vickers (HV) vs. time curves of the aged alloy are presented in Figure 3. The hardness slightly increases during aging at the indicated temperatures due to a low content of copper in the aluminium solid solution. The copper content in the aluminium solid solution in the commercial alloys is usually about 4–5% [22,24,25].

Figure 4 demonstrates the precipitate types in the aged alloy at 210 °C for 5 h after quenching. Three types of precipitates were identified in the thin structure by TEM investigation via EDX analyses. Two types,  $Al_{20}Cu_2Mn_3$  and  $Al_3(Zr,Er)$ , were nucleated during homogenization treatment at 605 °C and  $\theta''(Al_2Cu)$  during aging treatment at 210 °C. The  $Al_{20}Cu_2Mn_3$  phase has a length of 100–250 nm and a width of 70–120 nm. The average diameter of the  $Al_3(Zr,Er)$  precipitate phase is 35 nm. The rotation of Zr/Er in the  $Al_3(Zr,Er)$  phase is about 1/1 in accordance with point EDX analyses. The metastable disc shaped  $\theta''(Al_2Cu)$  precipitates with a thickness of 5 nm and a diameter of 100–200 nm were mostly nucleated on the  $Al_3(Zr,Er)$  precipitate phases (Figure 4). High resolution TEM image in Figure 4 demonstrates the  $\theta''(Al_2Cu)$  and  $Al_3(Zr,Er)$  phases.



**Figure 2.** Microstructure of the annealed alloy at 605 °C for (**a**) 1 h and (**b**) 3 h, and the distribution of alloying elements inside the white square on the SEM microstructure.



Figure 3. Hardness Vickers (HV) curves of the aged alloy at 150, 180, and 210 °C.



**Figure 4.** Microstructure of the annealed alloy at 605 °C for 3 h and the aged alloy at 210 °C for 5 h, and the EDX spectra from Al<sub>3</sub>(Zr,Er) and  $\theta''(Al_2Cu)$  particles (TEM).

The ingot of the investigated alloy was quenched at 605 °C after homogenization treatment for 3 h, rolled to a 1 mm thick sheet, and annealed at different temperatures. The HV vs. temperature and time curves of the annealed alloy after rolling are shown in Figure 5. Figure 5a demonstrates the evaluation of HV after 1 h of annealing at 100–550 °C in comparison with that of the Al-6Cu-4.05Er [19] and Al-4Cu-2.7Er-0.3Zr [30] alloys. The investigated alloy has a high hardness due to the addition of manganese. Two processes can take place during the annealing of the as-rolled investigated alloy: softening and strengthening. Softening during low temperature after rolling proceeds due to a decrease of the dislocation density and formation of the substructure with low angular boundaries. The fine particles of the Al<sub>3</sub>(Zr,Er) and Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phases retard softening. Strengthening is promoted due to aging processes and the nucleation of metastable  $\theta''(Al_2Cu)$  precipitates. A deformed structure with a high density of dislocations and vacancies accelerates strengthening. The HV peak was found after 1 h of annealing at 150 °C (Figure 5a) and after 2 h of annealing at 150 °C (Figure 5b). Strengthening by  $\theta''$ (Al<sub>2</sub>Cu) precipitates has a larger effect on material hardness than do softening processes at 150 °C. The hardness decreases by increasing the annealing temperature above 150 °C (Figure 5a). The hardness peak shifted to 0.5–1 h by increasing the annealing temperature up to 180 °C (Figure 5b). Softening has the largest effect on hardness at temperatures higher than 180 °C (Figure 5b). The recrystallization temperature of the alloy is in the range of 350-400 °C. A non-recrystallized grain structure was found after 1 h of annealing at 350 °C (inserted image in Figure 5a). The average grain size increased from 7.3 to 10.6  $\mu$ m by increasing the annealing temperature from 400 to 550 °C (inserted images in Figure 5a).



Figure 5. HV vs. (a) temperature and (b) time curves of the annealed alloy after rolling.

Tensile test samples were annealed at aging temperatures for different amounts of time. Results of the tensile tests are summarized in Table 2. The rolled yield strength (YS) is 344 MPa. Increasing annealing temperature from 150 to 210 °C leads to decreases in YS from 320–332 MPa to 290–298 MPa. The YS has the same value for up to 10 h of annealing. For example, the Al-4Cu-2.7Er-0.3Zr alloy has a YS of 268–274 MPa after annealing at 150 °C [30]. Alloying with Mg and Ti can further improve the strength of the investigated alloy.

Condition	YS, MPa	UTS, MPa	El., %
As rolled	$344 \pm 2$	$372 \pm 3$	$2.2 \pm 0.1$
Annealed at 150 °C for 2 h	$322 \pm 4$	$360 \pm 10$	$3.2 \pm 0.8$
Annealed at 150 °C for 6 h	$320 \pm 2$	$365 \pm 5$	$3.6 \pm 0.6$
Annealed at 150 °C for 10 h	$332 \pm 8$	$370 \pm 6$	$4.0 \pm 0.5$
Annealed at 180 °C for 1 h	$307 \pm 8$	$345 \pm 10$	$3.6 \pm 0.4$
Annealed at 180 °C for 6 h	$302 \pm 2$	$333 \pm 1$	$3.0 \pm 0.8$
Annealed at 180 °C for 10 h	$312 \pm 3$	$353 \pm 5$	$3.2 \pm 0.4$
Annealed at 210 °C for 1 h	$298 \pm 4$	$340 \pm 8$	$4.6\pm0.4$
Annealed at 210 °C for 6 h	$290 \pm 8$	$330 \pm 10$	$4.3 \pm 0.1$
Annealed at 210 °C for 10 h	$292 \pm 4$	$328 \pm 4$	$3.9\pm0.4$

 Table 2. Tensile tests results. YS: yield strength; UTS: ultimate tensile strength.

#### 4. Conclusions

The microstructure, phase composition, strengthening mechanisms, and mechanical properties of the novel Al-Cu-Er-Mn-Zr alloy were investigated. The quaternary (Al,Cu,Mn,Er) phase with atomic rotation of Cu/Mn/Er in 4/2/1 and possible composition of Al<sub>25</sub>Cu<sub>4</sub>Mn<sub>2</sub>Er was found in the as-cast alloy. The quaternary (Al,Cu,Mn,Er) phase did not change in size and morphology during homogenization treatment at 605 °C. The hardness slightly increased during aging at 150, 180, and 210 °C due to a low copper content in the aluminium solid solution. Three types of precipitates were identified in the structure. The Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> and Al<sub>3</sub>(Zr,Er) phase precipitates were nucleated during homogenization treatment at 605 °C and  $\theta''(Al_2Cu)$  phase precipitates were nucleated during aging treatment at 210 °C. The Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase has a length of 100–250 nm and a width of 70–120 nm. The average diameter of the Al<sub>3</sub>(Zr,Er) precipitate phase is 35 nm. The metastable disc shaped  $\theta''(Al_2Cu)$  precipitates with a thickness of 5 nm and a diameter of 100–200 nm were nucleated mostly on the Al<sub>3</sub>(Zr,Er) precipitate phases. The HV peak was found after annealing the rolled alloy at 150 °C due to strengthening by  $\theta''(Al_2Cu)$  precipitates, which have a larger effect on materials hardness than do softening processes. The novel Al-Cu-Er-Mn-Zr alloy has high tensile properties in the annealed alloy at 150 °C after rolling: YS = 320–332 MPa, ultimate tensile strength (UTS) = 360–370 MPa, and El. = 3.2–4.0%.

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