Effects of $\text{Al}_8\text{Cu}_4\text{Er}$ Phase on Corrosion Behavior of Al–Cu–Mg alloy with Er addition

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Abstract

The effect of $\text{Al}_8\text{Cu}_4\text{Er}$ phase on the corrosion behavior of Al–Cu–Mg alloy with rare earth Er addition in an under-aging state was studied. The results revealed that the addition Er into the Al–Cu–Mg alloy induced the formation of $\text{Al}_8\text{Cu}_4\text{Er}$ phase. The $\text{Al}_8\text{Cu}_4\text{Er}$ phase can significantly refine the grains during recrystallization, thereby suppressing the continuous precipitation of S-phase at grain boundaries and improving the resistance to intergranular corrosion. Conversely, the corrosion susceptibility of local regions around the $\text{Al}_8\text{Cu}_4\text{Er}$ phase with large dimension was higher than that of the AlCuFeMnSi phase, but weaker than that of θ phase, resulting in decreased pitting corrosion resistance.

Keywords Aluminum alloy · Er micro-alloying · Corrosion · Grain boundaries · Secondary phase

1 Introduction

Rare earth Er alloying is an important technology in modern industries [1]. The properties of aluminum alloys are associated with the microstructural evolution. Rare earth Er can modify their microstructures and affect their properties [2]. In the Al–20wt%Si alloy, appropriate Er addition can refine the primary Si and modify the structure of eutectic Si, resulting in improved ultimate tensile strength and elongation [3]. More importantly, the addition of Er creates a new Er-riched phase, which can affect various properties of aluminum alloys. In the 6063 aluminum alloy, Er alloying facilitated the formation of beneficial compounds (Al_8ErFe_4 and Er_5Si_3 phases) and reduced harmful elements, i.e., Si and Fe, hence improving the conductivity [4]. The Al_3Er phase, which typically appears in pure aluminum, as well as 5xxx and 7xxx aluminum alloys, is a popular new phase obtained by adding Er [5]. According to the Al–Er phase diagram, the

Al_3Er phase can form easily owing to the low eutectic point [6]. Furthermore, the Al_3Er phase with a melting point of 1067 °C is difficult to dissolve conventionally [7]. Fine and dispersed Al_3Er phases are coherent or semi-coherent with the matrix and act as heterogeneous nuclei during recrystallization nucleation, thereby increasing the recrystallization temperature and refining the grains [8]. Consequently, the mechanical properties can be enhanced considerably [9]. Furthermore, in the Al–Mg–Er alloys, increasing Er content reduced the amount of $\text{Al}_{66.7}\text{Mg}_{23.4}\text{Er}_{10}$ phase and increased the quantity of Al_3Er phase, which increased the electrochemical stability of passive films because the galvanic coupling of the Al_3Er -matrix exerted a less detrimental effect compared with the galvanic coupling of the $\text{Al}_{66.7}\text{Mg}_{23.4}\text{Er}_{10}$ -matrix [10]. It has been shown that other types of Er-riched phases rather than the Al_3Er phase in the 5xxx aluminum alloy can deteriorate the corrosion property [10]. Moreover, the tensile strength and elongation can be decreased [11]. The case illustrated that adding Er into Al–Mg alloy without solution treatment can form coarse Er-enriched constituents but no excess Er atoms to form Al_3Er phases; consequently, the mechanical properties deteriorated [11].

Hence, the Al_3Er phase is the rewarding particle to improve the property of aluminum alloys; however, this phase barely appears in 2xxx aluminum alloy [12]. In our simplified phase diagram of Al–Cu–Mg–Er alloy with 0.19 wt% Er addition (Fig. 1, calculated by FactSage

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Fig. 1 Simplified phase diagram of the Al–Cu–Mg–Er alloy with 0.19 wt% Er addition

thermochemical software), it is clear that the $\text{Al}_8\text{Cu}_4\text{Er}$ phase can be formed rather than the Al_3Er phase when the Cu content is about 4 wt%. During solidification, Er atoms primarily segregated at the grain boundaries and can more easily form the $\text{Al}_8\text{Cu}_4\text{Er}$ phase in the 2xxx aluminum alloy [13–15]. So how does the newly generated $\text{Al}_8\text{Cu}_4\text{Er}$ phase contribute to the strength and corrosion resistance of structural materials? A previous study has shown that the $\text{Al}_8\text{Cu}_4\text{Er}$ phase did not contribute to the strength [16]. However, the corrosion resistance regulated by $\text{Al}_8\text{Cu}_4\text{Er}$ phase has not been discussed hitherto. Moreover, for the 2xxx aluminum alloy, improving the corrosion resistance is important since it is applicable to harsh environments [17]. Therefore, studying the effects of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase generated by Er alloying on the corrosion behavior of the 2xxx aluminum alloy is necessary although the strength was not improved significantly.

Hence, the Al–Cu–Mg alloy was used as an experimental alloy in this study. Er was added into the Al–Cu–Mg alloy to study the effects of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase on the corrosion behavior. Intergranular corrosion (IGC), immersion, and electrochemical tests were performed to evaluate the corrosion property. Optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were employed for characterizing its microstructure.

2 Experimental

Ten kilograms of the Al–Cu–Mg alloy containing Er with thickness of 90 mm was prepared in a crucible furnace, and iron mold casting was performed. The casting temperature was 725 °C. The chemical compositions, detected by inductively coupled plasma atomic emission spectroscopy, are shown in Table 1. The as-received ingots were homogenized and subsequently rolled to 2 mm sheets. Solid solution treatment was performed at 495 °C for 1 h. Aging treatment was done at 190 °C for 1 h (under-aging) and 70 h (over-aging) [18]. Subsequently, these samples were tested for IGC according to the GB/T 7998-2005 standard. During tests, the samples were immersed in the corrosive medium of 10 mL H_2O_2 ($\rho = 1.10 \text{ g/mL}$) solution per liter of NaCl (57 g/L) solution for 6 h at $35 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$. Figure 2 shows the IGC sketch of the Al–Cu–Mg–Er alloys. The size of tested sample is shown in Fig. 2a. After the IGC tests were performed, the tested sample was cut perpendicular to the rolling direction (along the yellow face). The maximal depths (Fig. 2b) and morphologies of the IGC were analyzed by OM coupled with Image Pro software.

Open-circuit potential (OCP) tests were performed on a VersaSTAT 3F electrochemical workstation in an IGC test solution for 4 h. Potentiodynamic polarization tests were performed in 3.5% NaCl solution with the potential ranging from -0.9 to $-0.55 \text{ V}_{\text{SCE}}$ and the scanning rate of 1 mV/s. The electrochemical tests were tested at room temperature. Immersion tests were performed in 3.5% NaCl solution for 2 h and 3 h at room temperature.

Fig. 2 Intergranular corrosion sketch of the Al–Cu–Mg–Er alloys: **a** size of the tested samples; **b** maximal depth of the IGC

Table 1 Chemical compositions of the Al–Cu–Mg (–Er) alloys (wt%)

Alloys	Cu	Mg	Si	Fe	Mn	Ti	Er	Al
Al–Cu–Mg	3.88	1.46	0.25	0.11	0.6	0.075	0	Bal
Al–Cu–Mg–0.19Er	3.92	1.47	0.25	0.11	0.6	0.075	0.19	Bal
Al–Cu–Mg–0.34Er	4.02	1.50	0.25	0.11	0.6	0.075	0.34	Bal

The corrosion morphologies were observed by OM, and the precise depths of pits were analyzed after derusting by laser confocal microscope (Keyence, VK-X250) with a motorized z-axis stage [19]. The rust removal method is based on the GB/T 16545-2015 standard. TEM was applied to observe the grain boundaries and the secondary phases with an operating voltage of 200 kV. Electron-transparent regions were obtained by double jet electropolishing with an electrolyte comprising 30% nitric acid and 70% methanol below -30°C . SEM coupled with electron backscattered diffraction (EBSD) was employed to analyze the grain orientations and observe the secondary phase.

3 Results and discussion

3.1 Effects of Er addition on the IGC behavior

The secondary phases in the three under-aged alloys were analyzed, and the results are shown in Fig. 3. Two types of secondary phases are presented in the Al–Cu–Mg alloy (Fig. 3a), namely the AlCuFeMnSi (Fig. 3d) [20] and Al_2Cu (θ) phases (Fig. 3f) [21], which are the typical phases in the 2xxx aluminum alloy. In 0.19% Er alloyed Al–Cu–Mg alloy (Fig. 3b), the $\text{Al}_8\text{Cu}_4\text{Er}$ phase (Fig. 3e) was generated in solidification (Fig. 1) in addition to the two abovementioned secondary phases [21]. Regarding the 0.34% Er alloying

Fig. 3 Secondary phases in three under-aged alloys: **a** Al–Cu–Mg alloy; **b** Al–Cu–Mg–0.19Er alloy; **c** Al–Cu–Mg–0.34Er alloy; **d** composition of AlCuFeMnSi phase; **e** composition of $\text{Al}_8\text{Cu}_4\text{Er}$ phase; **f** composition of θ phase

(Fig. 3c), the types of secondary phase were similar to those of 0.19% alloying, but the dimensions of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase were larger.

The grain size of the Al–Cu–Mg–Er alloys was investigated, as shown in Fig. 4. The results show that the grain sizes were refined significantly (from 50.12 to 18.16 μm , Fig. 4a–c) with increased Er contents, which was mainly derived from the formation of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase (Fig. 3). The $\text{Al}_8\text{Cu}_4\text{Er}$ phase can pin the dislocation and subgrain to inhibit the grain growth process and elevate the recrystallization temperature [15]. Furthermore, the role of rare earth Er in grain refinement may also be that (but not the dominant factor in this study): (1) the rare earth Er can refine

the casting grain due to the solute segregation mechanism [13], which could associate with the grain refinement of under-aged alloys; (2) the trace amount of rare earth Er may increase the recrystallization temperature and stabilize the grain boundary, thereby refining the grains [22].

To reveal the effect of grain refinements on the precipitation during under-aging, the grain boundaries were observed, as shown in Fig. 5. Figure 5a–c shows the grain boundaries of the Al–Cu–Mg, Al–Cu–Mg–0.19Er, and Al–Cu–Mg–0.34Er alloys at the state of under-aging. The results show that a rod-like $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ phase (which is a T-phase [18, 23]) appears in the three alloys. The T-phase is produced during homogenization and cannot be removed

Fig. 4 Characteristics of microstructures in under-aged Al–Cu–Mg–Er alloys: orientation map of **a** Al–Cu–Mg, **b** Al–Cu–Mg–0.19Er, and **c** Al–Cu–Mg–0.34Er

Fig. 5 Grain boundaries of the Er-alloyed Al–Cu–Mg alloys: **a** under-aged Al–Cu–Mg alloy; **b** under-aged Al–Cu–Mg–0.19Er alloy; **c** under-aged Al–Cu–Mg–0.34Er alloy; **d** over-aged Al–Cu–Mg alloy; **e** composition of S-phase in **d**

by subsequent heat treatments [23]. Studies have shown that when the Cu/Mg ratio of the three alloys is 2.66–2.68, the main precipitate is the Al_2CuMg phase (S-phase) [24]. The size of S-phase precipitates is so small (nanoscale) that it cannot be observed in Fig. 3 using SEM. The composition of the precipitates could not be detected on the samples aged for relatively short times [25]; accordingly, after aging for 70 h (over-aging), the precipitates were randomly selected for composition examination. Figure 5d shows the precipitates at the grain boundary after aging for 70 h, and these precipitates can be regarded as the S-phase (Fig. 5e, as identified in our previous study [18]). Therefore, it was assumed that the continuous S-phases were decorated at grain boundaries of under-aged Al–Cu–Mg alloy (Fig. 5a). The corrosion susceptibility of the S-phase was significantly stronger than that of the T-phase [26] which was accidentally distributed at the grain boundaries (Fig. 5a). Therefore, this grain boundary characteristic indicates that the IGC susceptibilities are dominated by the S-phase at the grain boundaries. With the increase in Er content, the distribution of the S-phase at the grain boundaries changed from continuous to discontinuous (Fig. 5a and b). Subsequently, 0.34% Er alloying enlarged the gap between two S-phases at the grain boundaries (Fig. 5c), suggesting the improved IGC resistance of Er-alloyed Al–Cu–Mg alloy.

IGC tests were performed on the under-aged Al–Cu–Mg–Er alloys to confirm the microstructure reconstruction, the results of which are shown in Fig. 6. As shown in Fig. 6a, in the Al–Cu–Mg alloy, a rough network appeared after the IGC tests, and the maximal depth of the IGC reached 311.7 μm . When alloying 0.19% Er into the matrix, corrosion pits and IGC morphologies with a fine network were observed (Fig. 6b). The maximal depth of the IGC of Al–Cu–Mg–0.19Er alloy was 193.7 μm , which was significantly less than that of the Al–Cu–Mg alloy, indicating

that 0.19% Er alloying can improve the IGC resistance. As the Er content increased to 0.34%, the corrosion morphologies were unchanged in comparison with 0.19% Er alloying. IGC with a fine network and the corrosion of pits were also observed; the maximal depth of the IGC decreased to 180.2 μm (Fig. 6c). Therefore, it can be concluded that adding Er into the Al–Cu–Mg alloy can improve the IGC resistance.

As mentioned above, adding Er into the investigated Al–Cu–Mg alloy can form the $\text{Al}_8\text{Cu}_4\text{Er}$ phase (Fig. 3), which is corresponding to the simplified phase diagram of the Al–Cu–Mg–Er alloy (Fig. 1). Furthermore, the fragmented $\text{Al}_8\text{Cu}_4\text{Er}$ phase was distributed along rolling direction owing to the effect of rolling. During recrystallization, the $\text{Al}_8\text{Cu}_4\text{Er}$ phase refined the grains significantly (EBSD results, Fig. 4); therefore, the length of the grain boundary per unit volume is increased significantly, which could break the continuous S-phase and result in a more dispersed phase at the grain boundaries (Fig. 5) [27]. Finally, the preferable IGC resistance can be achieved by the Er alloying.

3.2 Pitting corrosion behavior with Er alloying

To further study the effect of Er alloying on the corrosion resistance of under-aged Al–Cu–Mg alloy, OCP and potentiodynamic polarization tests were performed, and the results are shown in Figs. 7 and 8. As shown in Fig. 7, with the increase in Er content, the OCP decreased gradually. The corrosion potential (E_{corr}) and corrosion current density (i_{corr}) obtained from the polarization curves (Fig. 8) are presented in Table 2. The results show that E_{corr} decreased and i_{corr} increased with increasing Er content, indicating that in the under-aging state, Er alloying can deteriorate other types of corrosion although the IGC resistance is improved.

Fig. 6 IGC morphologies of under-aged Al–Cu–Mg–Er alloys: **a** Al–Cu–Mg alloy; **b** Al–Cu–Mg–0.19Er alloy; **c** Al–Cu–Mg–0.34Er alloy

Fig. 7 OCP test results of Er-alloyed Al-Cu-Mg alloy in the under-aging state

Fig. 8 Potentiodynamic polarization test results of Er-alloyed Al-Cu-Mg alloy in the under-aging state

Table 2 Corrosion potential and corrosion current density obtained from the polarization curves shown in Fig. 8

Alloys	E_{corr} (mV)	i_{corr} ($\mu\text{A}/\text{cm}^2$)
Al-Cu-Mg	-613	4.21
Al-Cu-Mg-0.19Er	-610	5.19
Al-Cu-Mg-0.34Er	-624	9.56

Immersion tests in 3.5% NaCl solution for 3 h were performed to evaluate the pitting corrosion resistance of Er-alloyed Al-Cu-Mg alloy in the under-aging state, and the results are shown in Fig. 9. Small pits were dispersed in the Al-Cu-Mg alloy (Fig. 9a); however, the pit area enlarged with increasing Er content (Fig. 9b and c). The diameter of the pits evolved from several to tens of microns with increasing Er content. Furthermore, the

maximal pit depth and its average value (the tests were repeated thrice) were calculated, and the results are shown in Fig. 10. The maximal pits depth increased from 2.6 to 3.9 μm , and its average value increased from 1.5 to 3 μm with increasing Er content. Figures 9 and 10 indicate that adding Er into the Al-Cu-Mg alloy degenerated the pitting corrosion resistance, which corresponds to decreased OCP and E_{corr} , and increased i_{corr} .

Adding Er into the Al-Cu-Mg alloy decreased the OCP and E_{corr} , increased i_{corr} , and deteriorated the pitting corrosion resistance, which are related to the corrosion behavior of local regions around the secondary phases. Figure 11 shows the corrosion morphologies of the local region around the secondary phase in Al-Cu-Mg(-Er) alloy in the under-aged state after immersion tests in 3.5% NaCl solution were performed for 2 h. In the Al-Cu-Mg alloy, the local region at θ phase dissolved completely but that of AlCuFeMnSi phase underwent a slight corrosion dissolution (Fig. 11a). As shown in Fig. 3b and c, compared with the Al-Cu-Mg alloy, only the $\text{Al}_8\text{Cu}_4\text{Er}$ phase was the new phase presented in Er-alloyed Al-Cu-Mg alloy. Figure 11b shows the corrosion morphology of the local region around the $\text{Al}_8\text{Cu}_4\text{Er}$ phase in the Al-Cu-Mg-0.19Er alloy, and the results show that the matrix at the edge of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase underwent dissolution. Therefore, the abilities of the secondary phase to induce localized corrosion are ranked as follows: θ phase > $\text{Al}_8\text{Cu}_4\text{Er}$ phase > AlCuFeMnSi phase [28]. The depth of corrosion pits induced by secondary phases in the Al-Cu-Mg-0.19Er alloy is shown in Fig. 12. The results show that the corrosion depth induced by θ -phase (yellow arrow) was much smaller than that of $\text{Al}_8\text{Cu}_4\text{Er}$ phase (pink arrow), which indicates that the pitting corrosion resistance is dominated by the $\text{Al}_8\text{Cu}_4\text{Er}$ phase when alloying Er is added into the Al-Cu-Mg alloy.

The type of secondary phase was changed by adding Er, which decreased the pitting corrosion resistance. During solidification, the solute redistribution enabled the Er at the front of the solid-liquid interface to easily reach the eutectic composition point; therefore, the $\text{Al}_8\text{Cu}_4\text{Er}$ phase will nucleate and grow [13, 29, 30]. After heat treatment and rolling deformation, the $\text{Al}_8\text{Cu}_4\text{Er}$ phase was broken along the rolling direction (Fig. 3). With the increasing Er content, the dimensions of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase enlarged, although the other two secondary phases remained in the aluminum matrix. The abilities to induce localized corrosion were ranked as follows: θ phase > $\text{Al}_8\text{Cu}_4\text{Er}$ phase > AlCuFeMnSi phase. Furthermore, the dimension of θ phase is much smaller than that of $\text{Al}_8\text{Cu}_4\text{Er}$ phase, when undergoing corrosion, the pitting corrosion resistance was dominated by the $\text{Al}_8\text{Cu}_4\text{Er}$ phase. Therefore, Er-alloyed Al-Cu-Mg alloy exhibits the worse pitting corrosion resistance compared with the Al-Cu-Mg alloy owing to the corrosion of the local region around the newly generated $\text{Al}_8\text{Cu}_4\text{Er}$ phase.

Fig. 9 Immersion test results of Er alloyed Al–Cu–Mg alloy in the under-aging state: **a** Al–Cu–Mg alloy; **b** Al–Cu–Mg–0.19Er alloy; **c** Al–Cu–Mg–0.34Er alloy

Fig. 10 Maximal pit depth and its average value of Er-alloyed Al–Cu–Mg alloy at under-aging state

3.3 Relationship between IGC and pitting corrosion

Unlike other types of rare earth alloying [31–33], adding Er into the Al–Cu–Mg alloy induced the formation of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase, which is fundamental in regulating the IGC

and pitting corrosion. The $\text{Al}_8\text{Cu}_4\text{Er}$ phase can significantly refine the grains during recrystallization, which disperses S-phases at the grain boundaries and hence breaks the continuous corrosion channels and improves the IGC resistance [34]. Conversely, the local region at the newly generated $\text{Al}_8\text{Cu}_4\text{Er}$ phase has a relatively strong corrosion susceptibility and decreases the pitting corrosion resistance of Er-alloyed Al–Cu–Mg alloy. A previous study has shown that when adding rare earth Nd into the Al–5 Mg alloy, the pitting corrosion resistance was not significantly affected because the newly generated $\text{Al}_{11}\text{Nd}_3$ phase did not affect localized corrosion [35]. Recently, the addition of rare earth Sc and Zr to 2xxx series Al–Cu alloy was studied [36]. The results showed that the pitting corrosion resistance can be improved by adding Sc and Zr, as the sizes of the newly generated W-phase and $\text{Al}_3(\text{Sc}, \text{Zr})$ phase were refined although their number densities increased. Therefore, the characteristics [35] and size [36] of newly generated phase yielded by adding rare earth significantly affected the pitting corrosion resistance. In this study, the local region at the $\text{Al}_8\text{Cu}_4\text{Er}$ phase was sensitive to localized corrosion. Moreover, the dimensions of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase were much larger than those of the $\text{Al}_{11}\text{Nd}_3$ phase (generated by alloying Nd into Al–5 Mg alloy) [35] and W-phase (formed by alloying Sc

Fig. 11 Corrosion morphologies of local region around secondary phase in the Al–Cu–Mg(–Er) alloy in the under-aged state after immersion tests in 3.5% NaCl solution for 2 h: **a** Al–Cu–Mg alloy; **b** Al–Cu–Mg–0.19Er alloy

Fig. 12 Depth of corrosion pits induced by secondary phases in the Al-Cu-Mg-0.19Er alloy

and Zr into Al-Cu alloy) [36]; therefore, the pitting corrosion resistance of Er-alloyed Al-Cu-Mg alloy decreased.

As discussed above, the improvement in IGC resistance and the deterioration of pitting corrosion resistance is related to the generation of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase. The properties of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase can significantly affect the pitting corrosion resistance of the Al-Cu-Mg-Er alloy. By reducing the size of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase and rendering the distribution of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase more diffuse, not only can the grains be refined and the IGC resistance improved, but also the depth of pits reduced and the pitting corrosion resistance enhanced. Studies have shown that the appropriate heat treatment can dissolve the $\text{Al}_8\text{Cu}_4\text{Er}$ phase into $\alpha\text{-Al}$ [37], therefore, the suitable heat treatment may change the property of the $\text{Al}_8\text{Cu}_4\text{Er}$ phase and achieve excellent microstructures and corrosion resistance.

4 Conclusions

- (1) Adding Er into the Al-Cu-Mg alloy in the under-aging state decreased the maximal depth of IGC from 316.8 to 180.2 μm ; therefore, Er microalloying improved the resistance to IGC.
- (2) Adding Er into the Al-Cu-Mg alloy induced the formation of $\text{Al}_8\text{Cu}_4\text{Er}$ phase. The $\text{Al}_8\text{Cu}_4\text{Er}$ phase can refine the grains during recrystallization and thus the S-phase precipitation on the grain boundary was transformed from continuous to discontinuous during subsequent under-aging.
- (3) The ability of the secondary phase to induce localized corrosion was ranked as follows: θ phase > $\text{Al}_8\text{Cu}_4\text{Er}$ phase > AlCuFeMnSi phase. The size of $\text{Al}_8\text{Cu}_4\text{Er}$ phase was much larger than that of θ phase, and the pitting corrosion resistance was dominated by the $\text{Al}_8\text{Cu}_4\text{Er}$ phase. The local region between the alu-

minum alloy and newly generated $\text{Al}_8\text{Cu}_4\text{Er}$ phase body underwent dissolution and hence decreased the pitting corrosion resistance of Er-alloyed Al-Cu-Mg alloy.

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