



# Article Microstructure and Mechanical Properties of Novel Heat Resistant Cast Al-Cu-Yb(Gd)-Mg-Mn-Zr Alloys

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Abstract: The present study focused on the development of the novel heat resistant cast Al-Cu-Yb(Gd)-Mg-Mn-Zr alloys based on the prevue investigations. Microstructures and mechanical properties were investigated by optical, scanning and transmission electron microscopy, hardness measurements, and tensile and creep tests at room and elevated temperatures. Ytterbium in combination with Zr and Ti provide greater Al grain refining than gadolinium. The L1<sub>2</sub>-Al<sub>3</sub>(Zr,Yb) or L1<sub>2</sub>-Al<sub>3</sub>(Zr,Gd) and Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates were nucleated during solution treatment. The average sizes of  $L1_2$ -Al<sub>3</sub>(Zr,Yb) and  $L1_2$ -Al<sub>3</sub>(Zr,Gd) are 28 ± 6 nm and 32 ± 4 nm, respectively. Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates formed with a more coarse size of 100-200 nm. The highest hardening effect was achieved after 3 h of aging at 210 °C in both alloys due to S'(Al<sub>2</sub>CuMg) precipitates. The ultimate tensile strengths (UTS) of the AlCuYbMg and AlCuGdMg alloys at room temperature are 338 and 299 MPa, respectively. The UTS decreases to 220-272 MPa when increasing the temperature of the tensile test to 200-250 °C. The rupture stress at 250 °C for 100 h under stress is 111-113 MPa. The contribution from different structure parts in the yield strength was calculated. The main strengthening effects of 54–60 MPa and 138–153 MPa were achieved from  $L1_2$  and S' precipitates, respectively. The calculated values of yield strength (YS) are consistent with the experimental data. Novel AlCuYbMg and AlCuGdMg alloys are a potential option for castings for high temperature application.

Keywords: cast aluminum alloys; microstructure; mechanical properties; precipitates; heat resistance

## 1. Introduction

Aluminum alloys are the most popular lightweight materials for the automobile and aerospace industries due to their good combination of strength at room and elevated temperatures, density, casting properties and corrosion resistance [1,2]. Al-Cu-based cast alloys demonstrate a high strength and heat resistance but the worst casting properties, for example, high sensitivity for hot tearing [2–5].

There are several ways to improve the casting properties of Al-Cu-based alloys. Additional alloying by eutectic forming elements, such as Fe, Ni, Si, and Mn, provides an improvement in the hot tearing sensitivity, but the strengthening effect is lower [2,4,5]. Doping by trace amounts of rare earth metals (REM) as grain refiners, for example, yttrium, is a good way to improve the hot tearing resistance [6]. The most common way is to search the novel base systems to develop high technology Al alloys.

Recent studies have demonstrated a perspective of the ternary quasibinary system Al-Cu-REM, where REM = Ce, Y, Er, Yb and Gd, due to a narrow solidification range and high thermal stability of the intermetallic phases [7–17]. The mechanical properties of the ternary alloy can be effectively improved by Zr [18–21] and Mn [22–24] additions, which provide precipitation strengthening. Sequential alloying led to the development of novel heat



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). resistant casting and wrought alloys based on the Al-Cu-Y and Al-Cu-Er systems [25,26]. Novel alloys strengthen the eutectic phase particles, and L1<sub>2</sub>-Al<sub>3</sub>(Zr,REM) and Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates formed during solution treatment, and the S' (Al<sub>2</sub>CuMg) phase of the aging origin [18–26].

In addition, small additives of Yb or Gd may improve the mechanical properties and corrosion resistance of the Al-Cu-Mg and Al-Zn-Mg-Cu alloys [27–32]. For example, Yb refines the grains, decreases the precipitation temperature of  $\Omega$  phase, accelerates the aging hardening process, and increases the maximum hardness and the tensile strength of the extruded Al-Cu-Mg-Ag [22]. Ytterbium and gadolinium with zirconium provide an increase in the room and high temperature mechanical properties of the Al-Si-Mg alloys [33–35]. The main strengthening mechanism in the Yb- or Gd-containing alloys with Zr is L1<sub>2</sub>-Al<sub>3</sub>(Zr,REM), which precipitates nucleation [36–39]. The same strengthening effect should be achieved with scandium alloying but scandium is very expansive [40].

The present study focused on the development of the novel heat resistant cast Al-Cu-Yb(Gd)-Mg-Mn-Zr alloys based on the prevue investigations [17,21,24]. The microstructure evaluation during casting and heat treatment, which provide excellent mechanical properties at room and elevated temperatures, will be presented.

#### 2. Materials and Methods

Alloys with compositions presented in Table 1 were melted in the resistance furnace from pure Al (99.7%) and Mg (99.9%), and Al-10Yb, Al-10Gd, Al-10Mn, Al-5Zr, Al-5Ti-1B master alloys. Melting and pouring temperature was in the range of 780–800 °C. The master alloys with Mn, Zr, and Yb or Gd were successively introduced into the Al melt at 800 °C. Then pure Mg was introduced into the melt using titanium "bell". Al-5Ti-1B master alloy as a grain refiner was introduced in the melt before casting. Casting was carried out in the steel and copper water-cooling molds with cooling rate about 10–15 °C/s. The hot tearing index (HCI) was determined using "pencil" probe [2–5]. The average value of the HCI was calculated from three pourings.

Table 1. Chemical composition (wt.%) of the investigated alloys.

Alloy	Al	Cu	Yb or Gd	Mg	Mn	Zr	Fe	Si	Ti
AlCuYbMg	bal.	4.1	2.0	1	0.8	0.3	0.15	0.15	0.15
AlCuGdMg	bal.	4.5	2.7	1.1	0.8	0.3	0.15	0.15	0.15

Microstructure was investigated in detail with optical microscope (OM) Zeiss, scanning electron microscope (SEM) TESCAN VEGA 3LMH (Tescan, Brno, Czech Republic) and transmission electron microscope (TEM) JEOL-2100 EX (Jeol Ltd., Tokyo, Japan). Phase analysis was performed using X-ray diffraction (XRD) with Cu-K $\alpha$  radiation on a Bruker D8 Advance diffractometer. Chemical composition of the alloys was determined by SEM electron diffraction X-ray (EDX) analyses. The grain structure of as-cast samples was investigated using OM under polarized light. The microstructure was revealed by anodizing (15-25 V, 0-5 °C) using Barker's reagent (46 mL of HBF<sub>4</sub>, 7 g of HBO<sub>3</sub> and 970 mL of H<sub>2</sub>O). The average value of the grain size was measured by random secant method in 3 images. The specimens for TEM were prepared using the A2 electrolyte on Struers Tenupol-5 equipment. The solidus temperatures were determined by the Labsys Setaram differential scanning calorimeter (SETARAM Instrumentation, Caluire, France) (DSC). Ingots were solution treated at 555–565 °C for 3 h in the Nabertherm furnace. Aging treatment was carried out at 150, 180 and 210 °C in in the SNOL furnace. The hardness was measured by the standard Vickers method under 5 kg load. The hardness value HV was determined as the arithmetic mean of five measurements and the standard deviation was calculated. The tensile samples with diameter of 5 mm and gage length of 25 mm were stretched on a Zwick/Roell Z250 Allround (Zwick/Roell, Kennesaw, GA, USA) test machine. Three

samples were tested per condition. The rupture stress at 250  $^{\circ}$ C for 100 h under stress was determined on the Instron M3 test machine.

### 3. Results and Discussion

Zirconium and titanium elements are well known grain refiners in the Al alloys. The effect of grain refining should be increased in combination with other REM. For example, Er significantly refines the grain structure due to an increase in the nucleation centers [25,41–43]. The as-cast grain structures of the investigated alloys are presented in Figure 1. Ytterbium (Figure 1b) in combination with Zr and Ti provides greater refining than gadolinium (Figure 1a). The average grain sizes of the AlCuYbMg and AlCuGdMg alloys are  $60 \pm 12$  and  $100 \pm 15 \,\mu$ m, respectively.



Figure 1. As-cast grain structures of the (a) AlCuYbMg and (b) AlCuGdMg alloys (OM).

Prevue's study was targeted at the structures and properties of the same alloys without Mg, Fe and Si impurities [19].  $Al_{80-88}Cu_{8-12}Yb_{3-4}Mn$  and  $Al_{78-86}Cu_{10-15}Gd_{3-5}Mn$  phases of the solidification origin forms in the AlCuYbZrMn and AlCuGdZrMn alloys [24]. The same Mn-rich phases formed the eutectic microstructure in the investigated AlCuYbMg (Figure 2a,c) and AlCuGdMg (Figure 2b,d) alloys. In addition, Mn-rich phase particles with about 10% Mn were identified in the AlCuYbMg alloy. The formula of this phase can be written as  $Al_{22}Cu_3Mn_2Yb$ . Similar  $Al_{25}Cu_4Mn_2Y$  [22] and  $Al_{25}Cu_4Mn_2Er$  [23] phases were identified in the microstructures of the AlCuYZrMn and AlCuErZrMn alloys. Silicon impurity led to the  $Al_{80}Yb_5Cu_6Si_8$  and  $Al_{80}Gd_5Cu_8Si_5$  phase solidification in the AlCuYb [44] and AlCuGd [45] alloys. The volume fraction of the high Mn and Si-rich phases is very low (some peaks marked in the XRD patterns in Figure 2c,d). Magnesium in the investigated alloys led to the Mg\_2Si phase (black particles in Figure 2a,b) solidification. The as-cast composition of the Al solid solution is presented in Table 2.

Alloy	Al	Cu	Mg	Yb or Gd	Zr	Mn
AlCuYbMg	bal.	1.3–1.4	0.6-0.8	0.1-0.3	0.3–0.5	0.6-0.8
AlCuGdMg	bal.	1.2 - 1.4	0.8-0.9	0.1-0.3	0.4	0.6-0.8

Table 2. As-cast composition of the Al solid solution.

DSC curves of the AlCuYbMg and AlCuGdMg alloys are presented in Figure 3. The solidus temperatures of the Mg-free AlCuYbZrMn and AlCuGdZrMn alloys are 607 and 615 °C, respectively [24]. Formation of the Mg<sub>2</sub>Si phase in the investigated AlCuYbMg and AlCuGdMg alloys provides a decrease in the solidus temperature to 568 (Figure 3a) and 575 °C (Figure 3b), respectively.



**Figure 2.** As-cast microstructure and phase composition of the (**a**,**c**) AlCuYbMg and (**b**,**d**) AlCuGdMg alloys ((**a**,**b**)—SEM-BSE phase composition, (**c**,**d**)—XRD (grey lines—Mg-,Si- and Fe-free alloys)).



Figure 3. DSC curves of the (a) AlCuYbMg and (b) AlCuGdMg alloys.

The solution treatment temperatures of 555 and 565 °C for the AlCuYbMg and Al-CuGdMg alloys, respectively, were chosen in accordance with the measured solidus temperature. The microstructures of the AlCuYbMg and AlCuGdMg alloys after 3 h of solution treatment are presented in Figure 4. The intermetallic phase particles fragmentized, spheroidized and grew to 1–3  $\mu$ m. The non-equilibrium part of the intermetallic phases dissolved and provided an increase in the Cu and Mg content in the Al solid solution (compare the Tables 2 and 3). Fine white particles are clearly seen in the Al solid solution in SEM (Figure 4). A parallel process with solution treatment is the decomposition of the supersaturated Al solid solution by Zr, Mn and Yb or Gd.

Figure 5 demonstrates the TEM microstructures of the AlCuYbMg and AlCuGdMg alloys after 3 h of solution treatment at 555 °C and 565 °C, and quenching and aging at 210 °C for 3 h. The L1<sub>2</sub>-Al<sub>3</sub>(Zr,Yb) or L1<sub>2</sub>-Al<sub>3</sub>(Zr,Gd) and Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates were nucleated during solution treatment. Typical spherical precipitates with a coherent boundary with the Al solid solution (insert in Figure 4a) are homogenously distributed in

the microstructure (Figure 5). The average sizes of the L1<sub>2</sub>-Al<sub>3</sub>(Zr,Yb) and L1<sub>2</sub>-Al<sub>3</sub>(Zr,Gd) precipitates are  $28 \pm 6$  nm and  $32 \pm 4$  nm, respectively (Figure 4). For comparison, the precipitates sizes in the AlCuYbZrMn and AlCuGdZrMn alloys after solution treatment at 590 and 605 °C are  $38 \pm 10$  nm and  $45 \pm 16$  nm [19]. Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates formed with a finer size of 100-200 nm in comparison with the same particles in the Mg-free alloys [24].



Figure 4. Microstructures of the (a) AlCuYbMg and (b) AlCuGdMg alloys after 3 h of solution treatment at (a) 555 °C and (b) 565 °C (SEM-BSE).

Alloy	Al	Cu	Mg	Yb or Gd	Zr	Mn
AlCuYbMg	bal.	2.5-2.6	1.0-1.1	0.1-0.3	0.3–0.5	0.6–0.8
AlCuGdMg	bal.	2.0-2.2	1.1-1.2	0.1-0.3	0.4	0.6-0.8

Table 3. Composition of the Al solid solution after 3 h of solution treatment.



Figure 5. Microstructures of the (a) AlCuYbMg and (b) AlCuGdMg alloys after 3 h of solution treatment at (a) 555 °C and (b) 565 °C, quenching and aging at 210 °C for 3 h, and FFT pattern of L1<sub>2</sub>-Al<sub>3</sub>(Zr,Yb), and EDX spectrum from S' and Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phases (TEM).

AlCuYbMg and AlCuGdMg alloys were aged at 150, 180 and 210 °C after 3 h of solution treatment and quenching. HV vs. time dependences are presented in Figure 6. After being supersaturated by Cu and Mg, and after quenching, the Al solid solution decomposed with S'(Al<sub>2</sub>CuMg) precipitates nucleation. The highest hardening effect was achieved after 3 h of aging at 210 °C in both alloys due to S'(Al<sub>2</sub>CuMg) precipitates. Typical disk-shaped precipitates are identified in the TEM images in Figure 5.



Figure 6. Aging curves of the (a) AlCuYbMg and (b) AlCuGdMg alloys.

Table 4 and Figure 7 demonstrate the results of the tensile test and typical tensile curves at different temperatures of quenching and aging at 210 °C for 3 h alloys. The UTS of the AlCuYbMg and AlCuGdMg alloys at room temperature are 338 and 299 MPa, respectively. For comparison, commercial Al-5Cu-0.8Mn alloys have the same UTS of 313–334 MPa [2]. However, the casting properties of the novel alloys are better. The HCI of the AlCuYbMg and AlCuGdMg alloys is 12–14 mm but for commercial Al-5Cu-0.8Mn alloys it is closer to 16 mm [2–5]. The UTS decreases to 219–270 MPa with an increase in the temperature of the tensile test to 200–250 °C. At the same time the elongation significantly increases. The rupture stress at 250 °C for 100 h under stress is 95 MPa for a commercial 201.0 Al alloy [2]. Novel AlCuYbMg and AlCuGdMg alloys are a potential option for castings for high temperature application.

Table 4. Tensile test results at indicated temperature.

A 11 or .		20 °C			200 °C			250 °C		
Alloy	YS, MPa	UTS, MPa	El., %	YS, MPa	UTS, MPa	El., %	YS, MPa	UTS, MPa	El., %	
AlCuYbMg	$312\pm3$	$338\pm1$	$0.6\pm0.1$	$258\pm10$	$270\pm2$	$0.4\pm0.2$	$206\pm 6$	$219\pm 6$	$1.4\pm0.1$	
AlCuGdMg	$298\pm4$	$299\pm4$	$0.2\pm0.1$	$228\pm10$	$234\pm11$	$0.4\pm0.1$	$235\pm10$	$270\pm5$	$4.7\pm0.2$	

The yield strength (YS) of polycrystalline material is related to the critically resolved shear stress (CRSS) of the grains and the grain boundary strengthening [46–51]. In the present model, we consider five strengthening mechanisms that affect the CRSS of grains using a linear superposition:

$$\sigma_{\rm y} = \Delta \sigma_{\rm gb} + \Delta \sigma_{\rm d} + \Delta \sigma_{\rm ss} + \Delta \sigma_{\rm ppt} + \Delta \sigma_{\rm p} \tag{1}$$

where  $\Delta \sigma_{gb}$  and  $\Delta \sigma_d$  are the contribution from the grain boundaries and dislocations, respectively;  $\Delta \sigma_{ss}$  is the contribution from the solid solution;  $\Delta \sigma_{ppt}$  is the contribution from precipitates;  $\Delta \sigma_p$  is the contribution from eutectic particles.



Figure 7. Typical tensile curves of the AlCuYbMg and AlCuGdMg alloys at different temperatures.

The contributions from different structure parts are summarized in Table 5. The volume fraction of the precipitates was calculated from the Al-Zr, Al-Yb, Al-Gd, Al-Cu-Mg and Al-Cu-Mg-Mn phase diagrams. The main strengthening effects of 54–60 MPa and 138–153 MPa were achieved from L1<sub>2</sub> and S' precipitates, respectively. The calculated  $\sigma_y$  values are consistent with the experimental value of YS (Table 4).

Equation	Structure Parameters	AlCuYbMg	AlCuGdMg
$\Delta \sigma_{gb} = \sigma_0 + kd^{-0.5} [34-36]$	$ \begin{aligned} \sigma_0 &= 10 \text{ MPa,} \\ k = 0.065 \text{ MPa}/\text{m}^{-2} \end{aligned} $	18.4	16.5
$\Delta \sigma_{\rm d} = M \alpha_1 G b \sqrt{\rho_{\rm dis}} [34]$	$ ho_{\rm dis} = 109 \ {\rm sm}^{-2}$ [2]	21.2	21.2
$\Delta \sigma_{ss} = 13.8 C_{Cu} + 18.6 C_{Mg}$ [46]	$C_{Cu} = 0.12\%, C_{Mg} = 0.1\%$	3.5	3.5
Δσ <sub>p</sub> (Orovan equation [46])	r = 750 nm, f = 0.08	8.6	8.6
	L1 <sub>2</sub> (r <sub>Yb</sub> = 14 nm, r <sub>Gd</sub> = 16 mn, f = 0.007)	60.2	54.4
$\Delta \sigma_{ m ppt}$ (Orovan equations for spherical [46] and	$Al_{20}Cu_2Mn_3$ (r = 150 nm, f <sub>Yb</sub> = 0.0054, f <sub>Gd</sub> = 0.004)	14	12
disc shaped particles [50])	S'(Al <sub>2</sub> CuMg) ( $d_{Yb}$ = 200 nm, $d_{Gd}$ = 100 nm, h = 1.5 nm, $f_{Yb}$ = 0.04, $f_{Gd}$ = 0.037	153.9	138.6
σ.	279.8	254.8	

Table 5. Calculated contribution from different structure parts.

## 4. Conclusions

- 1. Ytterbium in combination with Zr and Ti provide greater refining than gadolinium. The average grains of the AlCuYbMg and AlCuGdMg alloys are  $60 \pm 12$  and  $100 \pm 15 \mu$ m, respectively.
- Al<sub>80-88</sub>Cu<sub>8-12</sub>Yb<sub>3-4</sub>Mn and Al<sub>78-86</sub>Cu<sub>10-15</sub>Gd<sub>3-5</sub>Mn phases of the solidification origin form in the investigated AlCuYbMg and AlCuGdMg alloys. In addition, Mn-rich phase particles with about 10%Mn were identified in the AlCuYbMg alloy. The formula of this phase can be written as Al<sub>22</sub>Cu<sub>3</sub>Mn<sub>2</sub>Yb. Magnesium led to the Mg<sub>2</sub>Si phase solidification.
- 3. The L1<sub>2</sub>-Al<sub>3</sub>(Zr,Yb) or L1<sub>2</sub>-Al<sub>3</sub>(Zr,Gd) and Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates were nucleated during solution treatment. The average sizes of the L1<sub>2</sub>-Al<sub>3</sub>(Zr,Yb) and L1<sub>2</sub>-

Al<sub>3</sub>(Zr,Gd) are  $28 \pm 6$  nm and  $32 \pm 4$  nm, respectively. Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> phase precipitates formed with a finer size 100–200 nm.

- 4. The highest hardening effect was achieved after 3 h of aging at 210 °C in both alloys due to S'(Al<sub>2</sub>CuMg) precipitates. Typical disk-shaped precipitates were identified in the TEM. The UTS of the AlCuYbMg and AlCuGdMg alloys at room temperature are 338 and 299 MPa, respectively. The UTS decreases to 220–272 MPa when increasing the temperature of the tensile test to 200–250 °C. At the same time the elongation significantly increases. The rupture stress at 250 °C for 100 h under stress is 111–113 MPa.
- 5. The contribution from different structure parts in the yield strength was calculated. The main strengthening effects of 54–60 MPa and 138–153 MPa were achieved from L1<sub>2</sub> and S' precipitates, respectively. The calculated values of YS are consistent with the experimental data.

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