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Creep behaviour of eutectic Zn-Al-Cu-Mg alloys

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ABSTRACT

In this study we investigated the creep behaviour of three eutectic ZnAl4Cu1Mg alloys with different Mg contents, namely ZnAl4Cu1Mg0.04, ZnAl4Cu1Mg0.21 and ZnAl4Cu1Mg0.31 (in wt%), using uniaxial tensile creep tests at temperatures between 25 °C and 105 °C. Furthermore, we studied the creep properties of the individual microstructural constituents of the ZnAl4Cu1Mg0.31 alloy using nanoindentation creep experiments at room temperature (25 °C) and 85 °C. Increasing Mg content causes an acceleration of the creep rate of ZnAl4Cu1 alloys. Zn-Al eutectic and eutectoid structures show a lower creep resistance than the η-Zn matrix phase. Stress exponents of 6.9–8.0 and creep activation energy values of 93–104 kJ/mol are obtained during the uniaxial tests, which suggests dislocation controlled creep.

1. Introduction

Zn-Al based alloys are attractive materials due to their physical and mechanical properties: high strength and wear resistance, excellent fluidity and castability, good surface finish as well as outstanding corrosion resistance [1–5]. Therefore, Zn-Al based alloys are widely used in the production of structural and decorative parts for automotive, architectural, electrical and electronic applications as well as machinery and equipment with a complex geometry or equipment needing a high manufacturing precision [4–9].

Due to their relatively low melting point (around 400 °C), Zn and Zn-Al based alloys are sensitive to creep. Tegart et al. [10] and Flinn et al. [11] have observed that the creep behaviour of pure zinc is controlled by two thermally activated processes acting additively. Specifically, at high temperatures ($T > 280$ °C), a creep activation energy, Q, of 152–168 kJ/mol [10–12], which is close to the activation energy of prismatic slip (152 kJ/mol [13]), has been observed. This suggests creep dominated by prismatic dislocation glide at temperatures above 280 °C [12,13]. At lower temperatures ($T = 40$–142 °C) a creep activation energy of 79–108 kJ/mol, which is close to the self-diffusion energy of Zn (92 kJ/mol [13,14]), has been reported, indicating that creep in this temperature range is controlled by the climb of edge dislocations [10,11,15]. Although Murthy et al. [16] have reported grain boundary sliding (GBS) during creep at temperatures between 80 °C and 200 °C in pure Zn, they have concluded that GBS is not the rate-controlling creep mechanism of pure zinc. Matsunaga et al. [17,18] have quantitatively measured the amount of grain boundary sliding during ambient-temperature creep in pure zinc using atomic force microscopy and found that grain boundary sliding accommodates about 30% of the total creep strain at a load of 19 MPa.

In contrast to pure Zn, various creep mechanisms have been reported for Zn-Al alloys. Generally, dislocation controlled creep has been reported for Zn-Al alloys at stress levels of 10–100 MPa and ambient temperatures [19–22]. Murphy et al. [19] have investigated the creep behaviour of three Zn-Al alloys with varying Al contents (4, 8, 27 wt%) and obtained a stress exponent $n = 3.5$–4.4 and a creep activation energy $Q = 106$ kJ/mol for stresses between 10 and 100 MPa at temperatures between 60 °C and 150 °C. Roberti et al. [20] and Kallien et al. [21] have studied the creep properties of a ZnAl4Cu1 alloy between 12 and 100 MPa and 25–85 °C. They have concluded that the ZnAl4Cu1 alloy has a stress exponent of 4–5 and an activation energy of around 94 kJ/mol, which indicates a self-diffusion controlled creep mechanism. In contrast, Alibabaie et al. [23] measured a much higher stress exponent ($n = 7.7$) and a lower activation energy of 52.5 kJ/mol at similar test conditions.

Further, Coble creep has been observed to be the predominant creep mechanism in Zn alloys with ultra-fine grain size [24] and in a Zn-22Al (wt%) eutectoid alloy at relative low stresses and high temperatures [25]. Gobien et al. [24] have studied the creep behaviour of an ultra-fine grained Zn-4.5Al alloy with an average grain size of 260 nm and determined a stress exponent of 1 and an activation energy close to that of grain boundary diffusion (60.5 kJ/mol [13]) in the temperature range of 22–100 °C. Prasad et al. [25] have reported a creep activation energy of 62 kJ/mol for a Zn-22Al (wt%) alloy at temperatures between 120 and 200 °C and stresses lower than 1 MPa. These activation energies [24,25] match well with that of grain boundary self-diffusion in

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Zn (60.5 kJ/mol [13]). Additionally, Nabarro-Herring creep has also been observed in a Zn-22Al (wt%) alloy at low stresses (0.1–0.3 MPa) and temperatures between 177 °C and 252 °C [26].

In general, zinc and zinc alloys possess different creep mechanisms which are sensitively determined by the temperature, stress and material state such as composition and grain size. A comprehensive summary of creep parameters for zinc and zinc alloys reported in the literature is given in Appendix (A).

Addition of reinforcement phases and / or alloying elements has been shown to improve the creep resistance of Zn alloys [24,27]. Particularly, second phase particles such as nitrides, oxides, silicon carbide (SiC) and alumina (Al_2O_3) have been reported to significantly reduce the creep rate of Zn-Al based alloys [24,27]. Similarly, addition of up to 1.2 wt% Cu to Zn-Al alloys has been reported to improve their creep resistance [21,28,29]. Hanna et al. [30] have attributed the improved creep resistance to the presence of the Cu-rich ε-phase. In contrast, Mahmudi et al. [30] and Anwar et al. [31] have reported that increasing Al content in Zn alloys results in higher creep rates and lower creep resistance. Finally, Mulvania et al. [29] have shown that Mn, Ti and Cr have essentially no effects on the creep strength.

It has been reported that Mg is effective in increasing the strength and ductility of Zn-Al alloys [5,7,8,32–36] through the modification of eutectoid structures [5] and formation of Zn-Mg intermetallic compounds [5,35,36]. However, the influence of Mg on the creep behaviour of Zn-Al-Cu alloys has not yet been comprehensively studied. Also, the role of the individual microstructural components in eutectic Zn-Al alloys during creep still remains unknown. Therefore, we investigated the creep mechanisms of ZnAl4Cu1Mg alloys with Mg contents of 0.04, 0.21 and 0.31 wt%, as well as the creep properties of the individual microstructural constituents.

2. Experimental methods

Three laboratory alloys, ZnAl4Cu1Mg0.04, ZnAl4Cu1Mg0.21 and ZnAl4Cu1Mg0.31, were prepared by gravity casting [5]. Table 1 gives the chemical composition of the alloys as determined by wet-chemical analysis [5]. The impurities such as Fe and Si were controlled to not exceed 10 ppm Si and 50 ppm Fe [5], since Mohamed et al. [37–39] and Uesugi et al. [40] reported that Fe and Si impurities can significantly influence the mechanical properties of Zn alloys. The mechanical properties of the as-cast zinc alloys were determined by tensile tests at 55 °C ($0.5\,T/T_m$), 85 °C ($0.55\,T/T_m$) and 105 °C ($0.58\,T/T_m$) at a strain rate ($\dot{\varepsilon}$) of $5 \cdot 10^{-4}\ s^{-1}$ using an electromechanical testing machine equipped with a furnace.

Uniaxial tensile creep tests were performed in air at 55 °C, 85 °C and 105 °C at stresses of $0.6{\cdot}\sigma_{0.2}$, $0.7{\cdot}\sigma_{0.2}$ and $0.8{\cdot}\sigma_{0.2}$, respectively, where $\sigma_{0.2}$ is the yield strength of the alloy at the corresponding temperature as determined by the tensile tests. The creep experiments were stopped when the stationary creep stage was reached. Table 2 lists the applied test parameters, where the normalised stress σ/G was calculated based on the corresponding shear moduli of the alloys (more details are given in Table 5). To obtain more reliable values for the stress exponent and activation energy, alloys ZnAl4Cu1Mg0.04 and ZnAl4Cu1Mg0.31 were additionally tested at 25 °C at stresses of $0.6{\cdot}\sigma_{0.2}$, $0.7{\cdot}\sigma_{0.2}$ and $0.8{\cdot}\sigma_{0.2}$.

Specimens for scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) observations were prepared by mechanical grinding and subsequent polishing down to an OPA (Al_2O_3 particles with a diameter of 0.03 µm) finish. The samples were examined with an FEI Helios Nanolab 600i SEM at an accelerating voltage of 20 kV using secondary electron (SE) microscopy and EBSD. Nanoindentation creep experiments were conducted at RT and 85 °C in vacuum ($6 \cdot 10^{-5}$ mbar) on a MicroMaterials NanoTest Platform 3 (modified for vacuum operation) at maximum loads of 15 mN, 25 mN and 35 mN using a cubic boron nitride Berkovich indenter. Frame stiffness and tip shape calibrations were carried out on fused silica and verified using a polycrystalline tungsten sample according to the method of Oliver and Pharr [41] before indentation. Loading was carried out at a constant strain rate of $0.1\ s^{-1}$, after which the load was held constant for 60 s and the change in displacement was continuously recorded. To minimize the thermal drift during indentation, the indenter and specimen were heated independently. During indentation experiments, the thermal drift was below $0.1\ nm\,s^{-1}$. At least 45 independent indentations were performed at each test condition.

Table 1
Chemical composition of the investigated zinc alloys.

Alloy	Element content, wt%			
	Al	Cu	Mg	Zn
ZnAl4Cu1Mg0.04	4.30	0.59	0.04	Bal.
ZnAl4Cu1Mg0.21	4.30	0.58	0.21	Bal.
ZnAl4Cu1Mg0.31	4.30	0.59	0.31	Bal.

Table 2
Applied parameters (temperature, stress) during uniaxial creep experiments[a].

Alloy		ZnAl4Cu1Mg0.04		ZnAl4Cu1Mg0.21		ZnAl4Cu1Mg0.31	
T, °C	$\sigma/\sigma_{0.2}$	σ, MPa	σ/G, $\cdot 10^{-3}$	σ, MPa	σ/G, $\cdot 10^{-3}$	σ, MPa	σ/G, $\cdot 10^{-3}$
55	0.6	87	1.89	98	2.14	87	1.90
	0.7	101	2.20	114	2.49	102	2.23
	0.8	115	2.50	130	2.84	117	2.56
85	0.6	76	1.69	93	2.07	80	1.79
	0.7	89	1.98	108	2.41	93	2.08
	0.8	101	2.25	124	2.77	106	2.37
105	0.6	61	1.38	65	1.47	63	1.43
	0.7	71	1.60	75	1.70	73	1.66
	0.8	81	1.83	85	1.92	83	1.88

[a] Additionally, alloy ZnAl4Cu1Mg0.21 was tested at 55 °C and 87 MPa and alloy ZnAl4Cu1Mg0.04 was tested at 85 °C and 93 MPa.

3. Results

3.1. Uniaxial tensile creep experiments

Fig. 1 shows the uniaxial tensile creep curves, namely the true strain vs. time curves, of the investigated ZnAl4Cu1Mg alloys obtained at 55 °C, 85 °C and 105 °C, at stresses of $0.6–0.8{\cdot}\sigma_{0.2}$. In this temperature and stress range, all samples reveal typical creep curves with primary and stationary creep stages. During the primary creep stage, the creep strain increases rapidly while the strain rate decreases upon loading. At the end of the primary creep stage, the creep strain increases almost linearly with time and the creep rate reaches a minimum, indicative of the second or stationary creep stage. Depending on the testing temperature, the samples enter the stationary creep stage at a creep strain ε of 0.4–1.4%. At the same temperature, the primary stage appears shorter at higher stress (e.g. Fig. 1.a), which is consistent with the observations of Arieli et al. [26] in a Zn-22Al (wt%) alloy. For some conditions (at high temperature and high stress level), the creep curves reach the tertiary stage within the testing time (e.g. Fig. 1.e), where the creep rate increases again rapidly until fracture.

Table 3 shows the time required to reach 1% creep strain for the investigated ZnAl4Cu1Mg alloys at different experimental conditions. Generally, the time to 1% strain decreases with increasing stress and / or increasing temperature. It is noticed that the time to reach 1% creep strain at 105 °C is longer than at 85 °C, which is assumed to be due to the higher normalised stress σ/G at 85 °C, Table 2. Additionally, under the same temperature and stress level, the time to reach 1% creep strain decreases with increasing Mg concentration in the alloy.

The creep rates of the secondary creep stage, i.e. the minimum or