Impression Creep of Mg-1.5Ca and Mg-3.0Ca Alloys

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Abstract: Mg-1.5Ca and Mg-3.0Ca alloys have been successfully synthesized using argon arc melting method. SEM and XRD were used to analyse the microstructure of the alloys. Creep resistance of the Mg-Ca alloys were investigated using impression creep technique at 423-523 K under constant stress of 357 MPa. Creep resistance for the Mg alloy enhanced with addition of Ca. Activation energies were calculated and found to be 142.5 and 108.4 kJ mol^{-1} for Mg-1.5Ca and Mg-3.0Ca samples, respectively. Microstructures in both alloys showed eutectic mixtures containing fine Mg$_2$Ca and α-Mg phases at both interdendritic and inter-grain regions.

Key words: Mg-Ca alloys, impression creep, mixtures, samples, regions

INTRODUCTION

Magnesium alloys are light-weight material, inexpensive and have high specific strength properties (Kainer, 2003). The applications of magnesium alloys in automotive components, such as gearbox housing, oil pan, transfer case, crankcase and tank coves which necessitate high creep resistance, have initiated deeper research in the particular area. Recent efforts to develop creep-resistant magnesium alloys for such applications have resulted in a number of experimental alloys (Horst and Mordike, 2006).

Among Mg alloys, Mg-Al based (AZ and AM series) alloys are widely used. These alloys possess high room temperature strength and ductility due to Mg$_{17}$Al$_{12}$. However, these alloys are prone to softening at elevated temperatures (>393 K) because of the low melting point of Mg$_{17}$Al$_{12}$ intermetallic phase (732 K) formed at the grain boundaries and interdentritic regions (Geranmayeh and Mahmudi, 2014). This phase has low thermal stability which leads to dissolution at temperatures higher than 398K and thereby decreasing the creep resistance (Jain and Koo, 2007; Mazraeshahi et al., 2013). Mg-Ca alloys were developed as Mg$_2$Ca has high melting temperature (988 K) and resist softening at elevated temperatures, hence increases creep resistance. Furthermore, Ca is a relatively inexpensive alloying element that can assist in grain refinement (Nayyeri and Mahmudi, 2010; Li at al., 2007; Yang and Li, 2013). It can also improve corrosion resistance, thermal and mechanical properties of magnesium alloys.

Impression creep test has attracted researchers as it is a relatively new tool to study the creep deformation behaviour of material. The method which uses cylindrical shape punch with a flat end, at constant load forced against flat surfaced samples has its advantages (Yang and Li, 2013). Impression creep test is a non-invasive test and only small amount of materials is sufficient for the test. Unlike conventional creep test whereby methods used often include measurement of plastic deformation at constant stresses or constant strain rate with respect to exposure of time, impression creep measures indentation depth with respect to indentation time (Gibbs et al., 1985).

During an impression creep test, the displacement of the cylindrical punch is monitored as a function of the elapsed time and the penetration rate or impression velocity can be estimated (Mathew and Vijayanand, 2013). A secondary steady state impression Velocity (V$_i$) is reached right after the primary transient period. During this stage strain hardening dominates compared to the recovery process. The slope of the straight line portion of displacement vs time impression creep plots gives the steady state impression velocity. This value of V$_i$ can be used to establish stress and temperature dependence of specific creep deformation mechanism (Chu and Li, 1977). In the case of polycrystalline metals and alloys dislocation creep and diffusion flow are the main deformation mechanisms involved in creep. Temperature (T) dependent steady state creep rate at constant microstructure and applied stress can be expressed as:

$$\bar{\varepsilon} = A_1 \exp\left(\frac{Q}{RT}\right) \qquad (1)$$

Where:
$\dot{\varepsilon}$ = The steady state creep rate
A$_1$ = A constant

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R = The universal gas constant. At high temperatures, activation energy

(Q) = For creep is determined by the active diffusion mechanism during deformation

The value of Q can be estimated by determining the slope of in $\acute{\varepsilon}$ vs 1/RT plot. As cast Mg alloys with addition of 4 wt% or more Ca have been reported to show reduced strength and fatigue strain (Gupta and Meenashisundaram, 2015). Hence, this paper reports the creep studies of two different compositions of Mg-Ca alloys, namely Mg-1.5 Ca and Mg-3.0 Ca, prepared by argon arc melting method. Experiments on creep resistance of the alloys were performed at temperature range of 432-523 K.

MATERIALS AND METHODS

Experimental: Two binary Mg-xCa (x = 1.5, 3.0 wt%) samples were prepared in this study. The nominal compositions are given in Table 1. The alloys were prepared using arc melting furnace on a water-cooled Cu heath in Ar atmosphere. The alloys were prepared with elemental metal with purities of 99.9% Mg and 70 wt% Mg-30 wt% Ca master alloy. The samples were metallographically prepared prior to etching using a solution of 2% nitric acid and 98% ethyl alcohol at room temperature. Microstructural examination of samples was conducted using a Zeiss Supra 40 VP Field Emission Scanning Electron Microscope (FESEM) equipped with attachment for Energy-Dispersive Spectroscopy (EDS). The phase identification was performed using a PANalytical X-ray diffractometer with Cu K$_a$ radiation. The measurements were conducted with step scan (2O) from 20-90° at increment of 0.02°. Impression creep testings of the samples were carried out using Wear and Friction Tech (Chennai, India) impression creep equipment at different temperatures (423-523 K). Furnace temperature control and impression load accuracy were within ±0.5 K and ±0.1 N, respectively. Cylindrical samples of 5mm height 15mm diameter were machined and surface was metallographically polished with 1 µm diamond slurry and three samples were tested for each condition. A flat end cylindrical tungsten carbide indenter with 1 mm diameter was used during this investigation and the data of indentation depth and time was obtained by displacement sensor (1 µm resolution) connected to computer by data acquisition hardware.

RESULTS AND DISCUSSION

The results from XRD analysis were indexed as shown in Fig. 1. XRD patterns of the samples show the

Table 1: Nominal composition of selected alloys

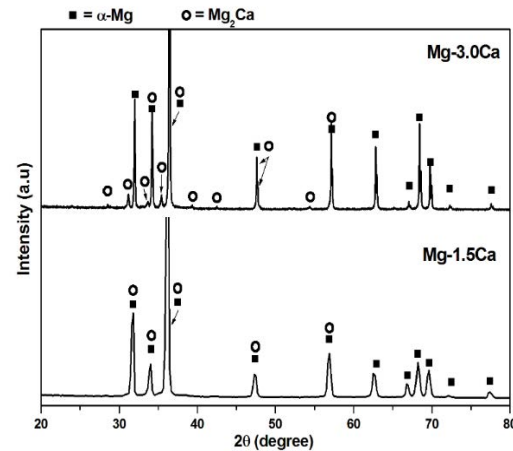| Alloy	Wt%	
	Mg	Ca (wt%)
Mg-1.5 Ca	1.5	Bal.
Mg-3.0 Ca	3.0	Bal.

Fig. 1: XRD patterns of Mg-1.5Ca and Mg-3.0Ca

presence of α-Mg and Mg$_2$Ca phases. SEM micrographs of the alloys are shown in Fig. 2. SEM micrographs showed primary Mg dendrites and eutectic mixture. The microstructural results are in agreement with results reported for Mg-Ca based alloy (Li *et al.*, 2008; Min *et al.*, 2011). The dendrites are seen to be more refined in Mg-3.0Ca compared to Mg-1.5Ca as can be seen from Fig. 2a, b. Higher magnification SEM micrographs as shown in Fig. 2c, d, revealed that eutectic mixtures contain fine Mg$_2$ Ca and α-Mg phases at both inter-dendritic and inter-grain regions. SEM micrographs of Mg-3.0Ca sample showed higher amounts of eutectic mixture compared to Mg-1.5Ca. Higher Ca in the eutectic mixture was confirmed by EDX analysis of both the phases.

Figure 3 shows the creep behaviour of (a) Mg-1.5 Ca and (b) Mg-3.0 Ca samples at various temperatures. All the curves show normal creep behavior, whereby short primary creep region is followed by steady secondary creep region. However, we cannot expect tertiary region because of the localized deformation due to indentation in compression mode. Strain hardening dominates the recovery process by dislocation climb during primary creep stage and they balance each other during secondary creep giving rise to steady state. Steady state impression velocities were obtained from the slopes of impression depth vs time curves of the steady state regions. Mg-3.0 Ca samples displayed lower creep deformation compared to the Mg-1.5 Ca samples at all the testing temperatures. Presence of higher amounts of
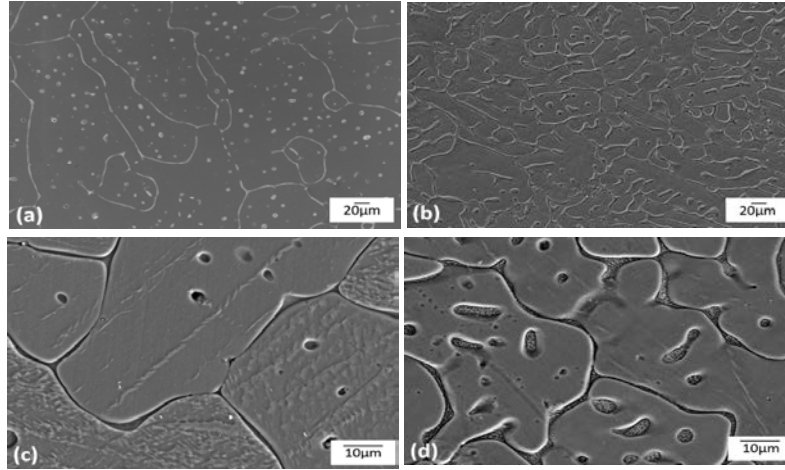
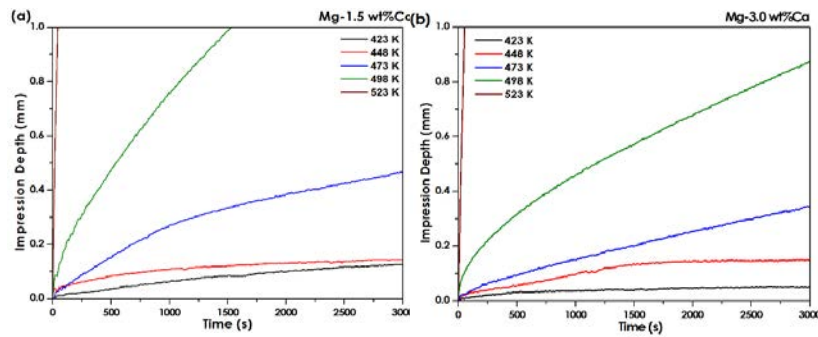Fig. 2: SEM micrograph of: a-c) Mg-1.5Ca and b-d) Mg-3.0Ca

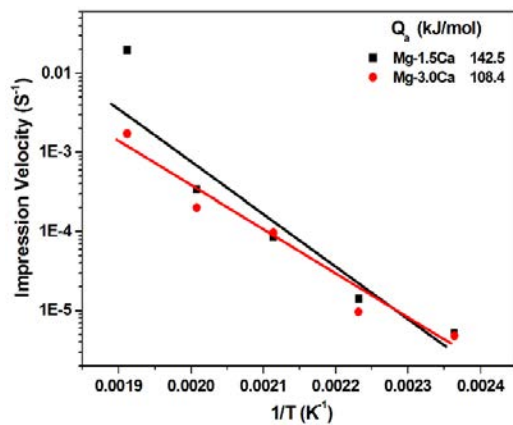Fig. 3: Creep at various temperature of (a) Mg-1.5Ca and (b) Mg-3.0Ca

Fig. 4: ln V_i vs 1/T at constant load of 157 MPa

Mg_2Ca in Mg-3.0Ca samples at inter-dendritic and inter-grain regions have offered higher resistance to deformation due to the inherent thermal stability of Mg_2Ca phase.

Activation energies for the dislocation climb during the creep deformation were estimated from the slopes of ln V_i and 1/T plots. The calculated activation energies are 142.5 and 108.4 kJ mol^{-1} for Mg-1.5Ca and Mg-3.0Ca samples, respectively (Fig. 4). The calculated activation energy for the Mg-1.5Ca sample is slightly higher than the lattice self-diffusion value for Mg (135 kJ mol^{-1}) suggesting that the dislocation climb is assisted by diffusion of Mg atoms in the bulk. Smaller activation energy values for the Mg-3.0 Ca sample suggest that other mechanisms such as grain boundary diffusion is also operative, apart from the lattice self-diffusion during dislocation climb during creep deformation. It is interesting to note that the grain size of the Mg-3.0Ca is smaller than the Mg-1.5 Ca sample and the higher grain boundary area could have contributed to the observed smaller activation energies for Mg-3.0 Ca samples.

CONCLUSION

From the results of this work it can be concluded that addition of Ca to Mg alloys resulted in grain refinement and reduced dendritic arm size as evident from SEM micrographs. Higher Ca in Mg-3.0Ca alloys has resulted in higher amounts of eutectic mixture containing Mg_2Ca phase which offered higher resistance to creep deformation at all temperatures compared to Mg-1.5Ca sample. Higher activation energy for Mg-1.5Ca (142 kJ mol^{-1}) sample suggests that creep mechanism is associated with dislocation climb assisted by self-diffusion of Mg. The contribution of grain boundary diffusion for dislocation climb is evident from the lower activation energy (108.4 kJ mol^{-1}) for Mg-3.0 Ca samples.

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