

UNIVERSITI TEKNOLOGI MARA

**EFFECTS OF Zr AND Sb ADDITION
ON GRAIN REFINEMENT, HIGH
TEMPERATURE CREEP AND
HARDNESS IN Mg-3Ca ALLOYS**

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ABSTRACT

Elemental addition is one method of improving the microstructure in alloys by grain refinement and consequently improving the mechanical properties such as creep resistance and hardness. Creep resistance of Mg alloys depends on the thermal stability of phases formed when alloying elements are added to Mg alloys. Otherwise, grain coarsening and dissolution at temperatures higher than 400 K may cause deterioration in creep properties. This thesis reports on the effects of Zr and Sb addition to as-cast Mg-Ca alloys prepared by argon arc melting. FESEM and XRD were used to analyze the microstructure of the alloys. The grain refinement effects and mechanism by adding Zr and Sb in Mg-Ca alloys and their influences towards creep resistance and hardness of Zr-added and Sb-added Mg-3Ca alloys at elevated temperatures are further discussed. Microstructures in Mg-1.5Ca and Mg-3Ca alloys showed eutectic mixtures containing fine Mg₂Ca and α -Mg phases at both interdendritic and inter-grain regions. Microstructural analysis of as-cast Mg-3Ca-*x*Zr (*x* = 0.3, 0.6, 0.9 wt.%) alloys showed grain refinement with Zr addition, attributed to the growth restriction effect of Zr in Mg-3Ca alloys. Addition of Sb to Mg-3Ca alloys also resulted in refined microstructure with formation of Mg₃Sb₂ in the interdendritic region. The grain refinement mechanism can be explained by the growth restriction factor (GRF) of the alloying elements in Mg. Rejection of solute into the melt at the solidification front of the solid/liquid interface caused subsequent release of latent heat, raising the temperature of the liquid between the interdendritic regions and allowed growth of dendritic arms. Creep resistance of the Mg-Ca alloys were investigated using impression creep technique at 423 – 523 K under constant stress of 380 MPa. Graphs of indentation depth against time determine the creep rate of samples. Activation energies calculated suggested mechanisms of creep deformation during impression creep tests. Creep resistance for Mg-3Ca alloys were enhanced with addition of Zr and Sb. Addition of Zr to Mg-3Ca alloys showed improved creep resistance due to solid solution strengthening of α -Mg by Zr. The activation energy value of Zr-added Mg alloys suggested that in Zr-added Mg-3Ca, the dislocation climb is assisted by both boundary and core diffusion. Sb addition to Mg-3Ca alloys displayed lower creep rate compared to Mg-3Ca alloy due to the presence of thermally stable Mg₂Ca and Mg₃Sb₂ in the interdendritic region. Q_c value of the samples with higher concentration of Sb suggests that the main mechanism during secondary stage creep is the dislocation climb. The hardness of Mg-3Ca-*x*Zr alloys increased as the amount of Zr increased due to grain refinement and solid solution strengthening of α -Mg by Zr. The addition of Sb increased the hardness of Mg-3Ca alloy by grain boundary strengthening and solid solution strengthening. The findings, analysis and discussions of the research in terms of grain refinement, high temperature creep deformation behaviour and hardness properties are summarized at the end of the thesis.

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CHAPTER ONE

INTRODUCTION

1.1 Background of Study

Magnesium (Mg) alloys are light-weight material, considerably less expensive compared to other alloys, and possess high specific strength properties (Kainer, 2003; Pekguleryuz, Kainer, Arslan Kaya, & Witte, 2013). Despite the excellent weight-to-strength ratio and room temperature mechanical properties, the range of applications for Mg alloys is associated with a number of limitations compared to other metals, such as aluminium alloys. These limitations include poor creep resistance at temperature more than 120 degrees Celsius (°C) or 393 Kelvin (K), lower tensile properties (strength and ductility), poor workability due to its hexagonal structure, high chemical reactivity, and low corrosion resistance (Ninomiya, Ojiro, & Kubota, 1995; Liu, Li, Wang, Su, & Han, 2009; Kabirian & Mahmudi, 2009; Ali, Qiu, Jiang, Pan, & Zhang, 2015). Accordingly, attempts have been made to develop new Mg based alloys having structural stability at high temperatures.

For example, the application of Mg alloys in automotive components such as gearbox housing, oil pan, transfer case, crankcase and tank coves, which necessitates high creep resistance, have initiated deeper research in the particular area. Recent efforts to develop creep resistant Mg alloys for such applications have resulted in a number of experimental alloys (Friedrich & Mordike, 2006). Continuous research is being pursued since the past decade to develop Mg alloys with other elemental additions with the aim of achieving refined microstructure, low density, high strength and creep resistant Mg alloys. One grain refinement technique for Mg alloys is the thermal–mechanical treatment upon solid Mg alloys, which leads to plastic deformation and dynamic recrystallization, and hence, small equiaxed grains can be obtained. Another approach is to achieve grain refinement during casting by stirring or through inoculation. Inoculation is considered as a successful approach to achieve grain refinement in Mg alloys. Inoculation by addition of grain refiners or small amounts of solute elements promote heterogeneous nucleation and restrict the grain growth (Ali et al., 2015).