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Correlation of grain boundary precipitate characteristics with fracture and fracture toughness in an Mg-8Al-0.5 Zn alloy

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ABSTRACT

In the present work, the fracture and fracture toughness properties of the Mg-0.8Al-0.5Zn alloy have been studied. In view of this, four different aging temperatures were selected to vary the size and area fraction of grain boundary precipitates and the associated precipitate-free zones (PFZs) width. The results suggest that the precipitation state at the grain boundary plays an important role in deciding the exact mode of failure (i.e., intergranular or transgranular type). The role of precipitate characteristics in determining the fracture mode has been ascertained. Moreover, the significance of preferential deformation within PFZ regions has been evaluated using TEM. The correlation between the various microstructural features and fracture toughness value is established following the Hornbogen and Graf model. The measured values of fracture toughness are found to be in good agreement with the values predicted by the model.

1. Introduction

Precipitation hardening is traditionally considered as one of the most important strengthening mechanisms for the development of high strength light weight alloys [1]. In particular, there has been significant work carried out on the precipitation strengthening in the Al alloys. Many authors [2–5] have reported the significance of precipitate characteristics in controlling the deformation and fracture behaviour of these alloys. The factors such as precipitate-free zones (PFZs), grain boundary precipitate size and area fraction are known to play a decisive role in regulating the fracture characteristics of Al alloys. In contrast, despite the advantage of weight savings with Mg alloys, there has been a very little effort in the development of age hardenable Mg alloys. This is primarily related to the fact that the conventional Mg alloys exhibit poor age hardening owing to a coarse precipitate distribution and its unfavourable orientation to block the slip [6,7]. Nonetheless, there has been a renewed interest developed in enhancing the age hardening response of Mg alloys by microalloying additions [1,6]. Factors such as grain boundary precipitate size, PFZs and precipitate distribution within the grain are likely to be important in controlling the mechanical behaviour of these alloys. An understanding of the effect of these variables on the fracture behaviour is important not only in developing the new alloys but also to enhance the performance of existing Mg alloys.

Several researchers [2–5,8–11] have investigated the effect of precipitate characteristics on the fracture behaviour of Al alloys. For instance, the work of Abe et al. [8] has emphasized the significance of localized deformation within PFZs in regulating the fracture characteristics of the alloy. Further, Unwin and Smith [5] have correlated the amount of intergranular fracture to the area fraction of grain boundary precipitates. Their results indicate that the proportion of intergranular fracture decreases with a decrease in the area fraction of precipitates. However, their results do not show any significant variation in the tensile properties with the change in PFZ width. For an Al-Li alloy, Vasudevan and Doherty [9] have reported that fracture toughness varies with the inverse square root of area fraction of grain boundary precipitates at constant yield strength. They suggested that the large grain boundary precipitate is more harmful for the initiation of grain boundary ductile fracture at a constant areal fraction of grain boundary precipitates than the smaller size precipitates. However, their results do not show any dependence of PFZ width on the fracture toughness. Similarly, Embury and Nes [4] also found a relationship between the fracture toughness and area fraction of grain boundary precipitates in Al-Zn-Mg alloys. As compared to above, limited research has been carried out on Mg alloys, where the characteristics of grain boundary microstructural features are correlated with the deformation and fracture behaviour. For instance, Zheng et al. [12] have correlated the dominance of fracture mode (i.e., intergranular or transgranular) with

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the grain boundary precipitates and PFZ for Mg-Gd-Nd-Zr alloy system. They suggested that larger grain boundary precipitates and wider PFZ width promote the tendency of intergranular fracture, whereas the transgranular cleavage fracture is dominated at smaller precipitate size and PFZ width. Yu et al. [13] also mentioned about the significance of grain boundary precipitates and PFZs in promoting the intergranular fracture in Mg-Zn-Mn alloy.

In this study, the effect of grain boundary precipitates on the fracture and fracture toughness behaviour of AZ80 Mg alloy has been evaluated. Four different microstructures, exhibiting different precipitate characteristics, have been produced by varying heat treatment conditions. Fractography and transmission electron microscopy (TEM) are utilised to investigate the fracture behaviour. The impact of PFZ width and grain boundary precipitate size and area fraction on the fracture characteristics has been examined. The significance of these microstructural features on the fracture toughness of AZ80 alloy has also been evaluated using Hornbogen and Graf model [14].

2. Experimental procedure

2.1. Material and microstructure analysis

In this study, as-cast AZ80 magnesium alloy (8 wt% Al- 0.5 wt% Zn) was used as a starting material. Samples of 10 mm × 9 mm × 8 mm were cut from an as-cast ingot using wire electric discharge machining (EDM). These samples were solutionized at 390 °C for 24 h, quenched in water and then immediately aged at four different temperatures (200, 250, 300 and 330 °C) for 48 h. The aging behaviour of AZ80 alloy was determined by measuring the micro-hardness using Vickers micro-hardness tester (HMV2, Shimadzu) with a load of 3 N for the dwell time of 10 s. Fig. 1 shows the variation of micro-hardness for peak-aged and over-aged samples at different aging temperatures. It should be noted here that the aging conditions used in this study correspond to an over-aged state. The over-aged condition was chosen as it provides a sufficient variation in grain boundary precipitate characteristics and PFZ width with a little change in the overall yield strength. It can be seen in Fig. 1 that the hardness of the alloy decreases with an increase in aging temperature. Moreover, aging time corresponding to the peak hardness decreases with an increase in aging temperature. For instance, the time to achieve peak hardness at 200 °C was ~ 24 h, whereas peak hardness was achieved in only 8 h in the case of 330 °C. These variations are consistent with the previous work [7,15].

For microstructural characterization, these samples were first mechanically polished using four different grades of silicon carbide emery papers (320, 1200, 2500 and 4000 mesh size) followed by the cloth polishing with silica suspension of 0.05 µm. To reveal the microstructure, the samples were first chemically etched with 10% Nital and subsequently with 10% HF solution. FEG SEM (FEI Quanta 200F SEM) was used to characterize the microstructures in terms of PFZ and precipitates. For examining the dislocation structure within PFZs and inside the grains, TEM observations were made by using JEM-2100 F transmission electron microscope. For TEM sample preparation, disks of 3 mm diameter were mechanically ground to ~ 70 µm in thickness. In the final step, the samples were ion milled to perforation at an ion accelerating voltage of 5 kV. The PFZ width was measured across the grain boundary (from one grain to another), covering the PFZ in two grains, as depicted in Fig. 2. To determine the average width of PFZ, about 10 grain boundaries were chosen from different micrographs and for each grain boundary ~ 10 random distances were measured. The average size of grain boundary precipitates (long dimension, h), shown in Fig. 2, was measured by considering ~ 100 precipitates from different grain boundaries. The area fraction A_f of grain boundary precipitates was estimated based on the relationship given by Embury and Ness [4] as:

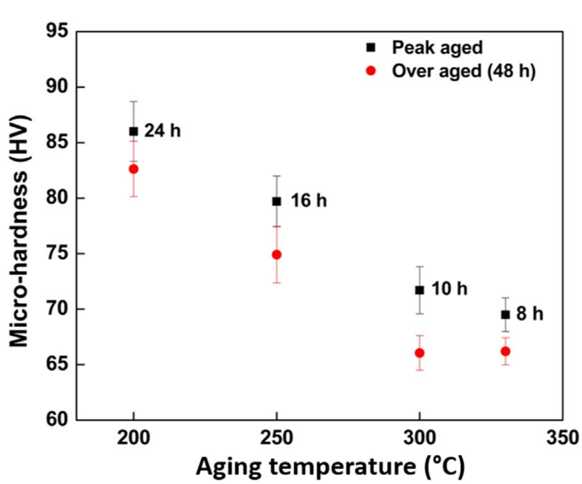

Fig. 1. The variation of hardness with aging temperature for peak-aged and over-aged conditions.

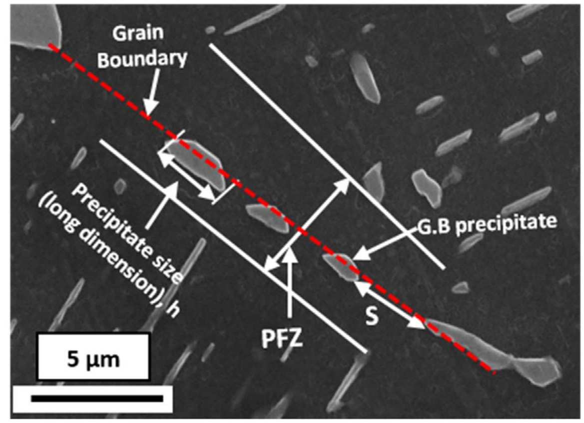

Fig. 2. Illustration of a PFZ region adjacent to a grain boundary and associated measurements of various precipitate dimensions.

$$\text{Area fraction,}\quad A_f = \left(\frac{h}{h+s}\right)^2 \tag{1}$$

where, h is the length of a grain boundary precipitate and s is the surface to surface distance between the neighbouring precipitates lying on a single grain boundary (Fig. 2).

2.2. Tensile testing

Uniaxial tensile test was performed at room temperature on the 30 kN capacity screw driven Zwick tensile machine at a constant strain rate of 0.001 s^{-1}. Tensile test specimens were prepared using wire EDM according to ASTM: E8/E8M standard [16]. Prior to tensile testing, samples were also polished to examine the effect of the microstructural features on the fracture behaviour. For each aging condition, at least three tests were performed to ensure the repeatability. To measure the elongation, a video extensometer (non-contact mode) was attached to the machine. The yield strength was estimated by applying the 0.2% offset on the stress-strain curve. The values of strain hardening exponent (n_i) were determined by using the Hollomon relationship which is given as $\sigma_T = K\epsilon_T^n$, where σ_T is the true stress, ε_T is the true strain and K is the strength coefficient [17]. Fractography was performed post tensile test using FEG SEM.

2.3. Fracture toughness testing

Fracture toughness test was performed on MTS 880 universal testing machine with a constant cross head speed of 0.5 mm/min. Specimens