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Short Communication

A closer inspection of a grain boundary immune to intergranular corrosion in a sensitised Al-Mg alloy

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A B S T R A C T

The intergranular corrosion of Al-Mg alloys arises from sensitisation due to thermal exposure. Here, a protected low angle (8.7°) grain boundary which hinders the intergranular corrosion propagation in a sensitised (150°/7 d) Al-Mg alloy was studied in detail. Discrete β-phase was observed on this protected boundary, but with a lower continuity compared to the other fully (or partially) dissolved grain boundaries. The results herein seek to unify the relationship between β-phase continuity and grain boundary misorientation angle as critical for intergranular corrosion propagation in the studied alloy.

1. Introduction

The 5xxx series aluminium (Al) alloys are based on the Al-Mg(-Mn) system, and are widely used for marine applications where they provide general corrosion resistance and weldability, in addition to moderate strength [1]. Most of the 5xxx series alloys are cold rolled and solid solution strengthened with a supersaturation of magnesium (Mg) that has been quenched into solid solution. (i.e. alloys such as AA5083 contain > 3.5 wt.% Mg) [2,3]. However, 5xxx series Al-alloys are susceptible to intergranular corrosion (IGC) and intergranular stress corrosion (IGSCC) upon prolonged exposure to elevated temperature [4,5]. Such IGC occurs owing to the precipitation of β-phase (Mg_2Al_3) via decomposition of the saturated solid solution, in particular precipitates tend to initially form at grain boundaries; a process that is termed sensitisation [6–9]. Attempts to model the precipitation of grain boundary (GB) β-phase has recently been undertaken in some detail [10], The β-phase is a so-called anode when in the Al-alloy matrix [11], and local self-dissolution of β-phase dissolution results in severe IGC or IGSCC damage [12,13].

A number of studies have provided evidence regarding the influence of grain boundary character on the sensitisation of 5xxx series Al-alloys [5,14–19], whereby low angle grain boundaries (LAGBs, defined as having a grain to grain misorientation of < 15°) have been reported to be more IGC resistant [5]. In such cases, it has been hypothesised that an absence of grain boundary β-phase may be responsible for the IGC resistance at LAGBs, a hypothesis supported from the decreased DoS in sensitised Al-Mg alloys with higher fraction of LAGBs [19]. However, there are recent studies that have revealed that β-phase may also nucleated on LAGBs [18,20], in contrast to any prior assertions that LAGBs may be free of β-phase [17]. It however merits comment that much of the work to date on the sensitisation of 5xxx series Al-alloys has evolved from optical microscopy of etched specimens. In such studies, attacked grain boundaries are assumed to be decorated with β-phase, whilst unattacked grain boundaries are assumed to be free of β-phase; the latter assumption however is not entirely accurate. An unattacked GB is indeed unattacked, but it may still contain a population of β-phase, the identification of which requires specific inspection at the nanoscale. The present study seeks to definitively isolate the characteristics of an IGC immune GB in sensitised Al-Mg-Mn alloy, in order to address a finite knowledge gap between the microstructural characterisation and IGC test performance. Herein, we apply transmission Kikuchi diffraction (TKD) and scanning transmission electron microscopy to identify and characterise IGC immune GBs.

2. Experimental

The Al-Mg-Mn alloy studied herein was typical of the composition of AA5083, and prepared for this study. It was produced from 99.9% pure Al, Mg and Mn (supplied by Alfa-Aesar). The alloy was produced by melting Al in a graphite crucible (750 °C for 40 mins) followed by the addition of Mg and Mn into the molten Al pool. The melt was repeatedly stirred throughout a further 750 °C hold, to achieve a homogenous mixture prior to casting into a 200 °C pre-heated steel mould. The casting was homogenised at 450 °C for 6 h, followed by water quenching. Hot rolling was then performed at 450 °C with 50% reduction, followed by water quenching. The as-rolled samples were

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Table 1
Composition of the Al-Mg alloy studied in this work (determined by ICP-AES).

	Mg	Mn	Fe	Si	Cr	Cu	Zn	Ti	Al
wt.%	3.80	0.25	0.11	0.04	0.03	0.01	0.01	0.01	Bal.

recrystallised at 450 °C for 10 mins and water quenched, followed by cold-rolling at room temperature with 20% reduction in sample thickness. The final composition of the alloy was obtained from inductively coupled plasma – atomic emission spectroscopy (ICP-AES, Spectrometer Services, Coburg, VIC, Australia), is shown in Table 1.

The alloy specimen was sensitised at 150 °C for 7 days, and air cooled, resulting in a degree of sensitisation (DoS) of 34.86 ± 7.7 mg/cm^2. The DoS was determined by the nitric acid mass loss test (NAMLT) method according to ASTM G67-04, and elaborated elsewhere [21]. Following NAMLT testing of the sensitised specimen, a grain boundary that was determined to be immune to intergranular corrosion, was identified with the aid of scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) (JEOL – 7001® FEG-SEM equipped with Oxford instruments AZtec® X-ray analysis system and HKL EBSD analysis).For EBSD analysis, all specimens were prepared metallographically to 0.05 μm finish, followed by ion milling using GATAN precision etching coating system (PECS™). A thin lamella containing the immune grain boundary was sectioned using a focused ion beam lift-out procedure (FEI Quanta® 3D FEG, equipped with a Pegasus Hikari® EBSD system). To examine the GB misorientations of the lamella, the transmission Kikuchi diffraction (TKD) technique was applied. The lamella was also studied by the scanning transmission electron microscopy (STEM) using an FEI Tecnai F20 operated at 200 kV.

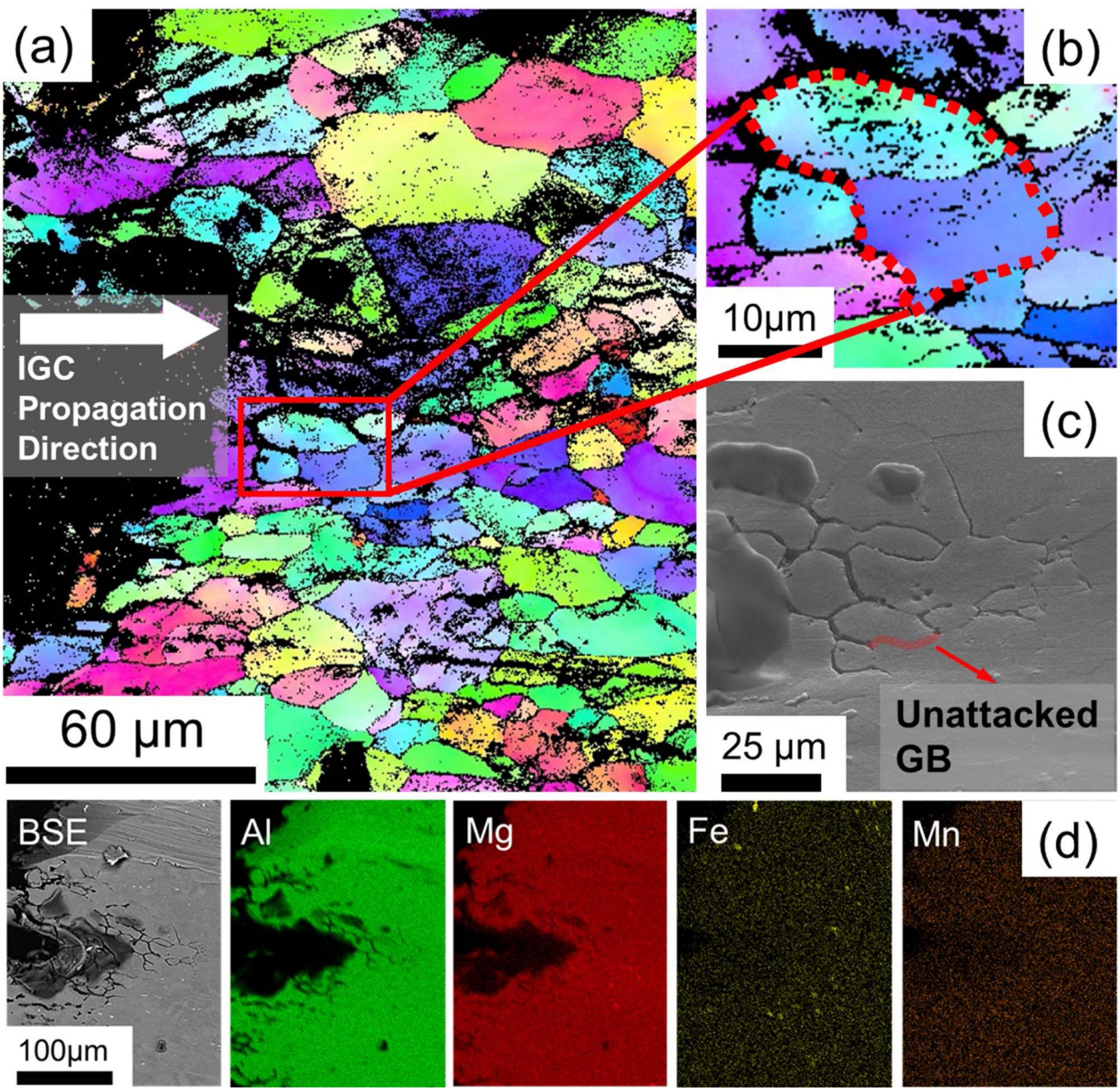

Fig. 1. The cross-section of a sensitised and NAMLT tested sample (DoS = 34.86 mg/cm^2) characterised with EBSD at (a) low, and (b) medium magnification. The SEM, BSE and EDS images of the same area are presented in (c) and (d). It should be noted that the intergranular corrosion tortuously propagated from left (the surface exposed to concentrated nitric acid in the NAMLT test) to the right (inwards to the matrix).