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Combined nuclear microprobe and TEM study of corrosion pit nucleation by intermetallics in aerospace aluminium alloys

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Abstract

A microscopic study of the onset of pitting corrosion has been conducted on the important aerospace alloy AA2024-T3 with the aim of understanding the role of intermetallic particles in triggering the formation of deep pits in the corroding surface. By trace element and correlation mapping using a 3 MeV H^+ microprobe, it has been shown that Cu–Fe–Mn–Al particles in conjunction with S-phase particles have a high correlation with pit nucleation. Al–Cu correlation diagrams can be used to show dealloying of S-phase particles ($CuMgAl_2$). Transmission electron microscopy and elemental mapping were used to look at the distribution of elements, particularly Cu, within pits.
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1. Introduction

Aluminium alloys for commercial applications contain numerous intermetallic inclusions comprising of one or more of the following major elements: Al, Fe, Cr, Si, Cu and Mn [1]. These inclusions typically take on many shapes and have diameters up to 50 μm on a rolled surface [2]. Particle densities ranging from 3×10^5 to $1 \times 10^6/cm^2$ have been measured [2–4]. Exposed surfaces of these alloys require environmental protection against corrosion, which is usually accomplished by means of a conversion coating using Cr compounds or anodising followed by a paint system

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which may include a chromate inhibited primer and topcoat. The breakdown of these coatings allows the onset of corrosion in the aluminium alloy which is a significant problem given the wide application of aluminium alloys in infrastructure and transport (particularly aerospace).

Pitting corrosion in aluminium alloys is particularly important since pitting can lead to other forms of corrosion such as exfoliation corrosion which undermines structural integrity [5]. Pitting corrosion is localised and it has been demonstrated that intermetallics play an important role in pit formation [3,4]. The main focus of this paper is to investigate the presence of different intermetallic inclusions and correlate their locations with points where pitting corrosion has initiated in AA2024-T3. The aim is to understand the mechanisms behind corrosion initiation and propagation. Due to the small size of the pits, high spatial resolution analysis techniques are required. The elemental distributions around the pits were mapped using a nuclear microprobe and particle induced X-ray emission (PIXE) analysis. For comparison, cross sectional transmission electron microscopy (X-TEM) was also performed.

2. Experimental method

2.1. The alloy samples

The Al-alloy AA2024-T3 [5] belongs to the 2XXX series of Alloys (Al–Cu–Mg) and was the first of this type have a yield strength approaching 490 MPa [5]. Its desirable properties include high strength and fatigue performance [5]. It is used in aerospace applications like aircraft fuselage, wing skins and engine areas. The main alloying elements are Cu (3.8–4.9%) and Mg (1.2–1.8%) it also contains Si (0.5%), Fe (0.5%), Mn (0.3–0.9%), Cr (0.1%), Zn (0.25%) and Ti (0.15%).

Polished substrates were prepared by pressing out discs of AA2024-T3 sheet 1.6 mm in thickness and then ground using silicon carbide papers to P1200, before final polishing down with 0.25 μm diamond. Corrosion was initiated by placing the polished specimens in either an aerated 0.1 M NaCl or 0.1 M NaCl solution which had 4×10^{-4} Molar $K_2Cr_2O_7$ solution added after pits had been initiated. After an induction period, corrosion in the form of pits with hydrogen evolution was observed on the surface.

2.2. Nuclear microprobe analysis

Both polished and NaCl corroded samples were exposed to a 3.0 MeV proton beam. Particle induced X-ray emission (PIXE) measurements were performed using an 80 μm thick Be filter (to reduce the aluminium signal) and was placed in front of the Ge X-ray detector which was located 165 mm from the sample stage. Analysis and phase correlation mapping of respective elements located at pit sites were performed using GeoPIXE [6].

2.3. TEM analysis

X-TEM samples, of nominally 100 nm thickness, were prepared by ultramicrotomy using a diamond knife with a 35° cutting edge. These samples were analysed using a JEOL 2010 transmission electron microscope fitted with a Gatan imaging filter (GIF). Energy filtered elemental maps were collected with the GIF using the three window method (two for the background and one for the characteristic edge). The resultant intensity for each pixel was then plotted as an elemental map. Processing of the elemental maps was performed using Gatan's Digital Micrograph 3.8.2 software.

3. Results and discussion

3.1. PIXE on polished AA2024-T3

Fig. 1 shows Al, Mn, Fe and Cu PIXE elemental maps of the polished AA2024-T3 alloy. Intermetallic particles are clearly visible on the surface. The PIXE analysis reveals two main types of intermetallic particles. Circles in Fig. 1 show particles approximately 20 μm which contain high levels of Mn, Cu and Fe. These particles correspond to the Cu–Fe–Mn–Al type intermetallic. According to Buchheit et al. [3] intermetallics in this alloy include 61.3% $CuMgAl_2$ (S-phase), 12.3% Al_6(Cu, Fe,

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