TEM Sample Preparation Method for Grain Boundary Phase Identification in Al-Mg alloys

CLASS 3: Electron Microscopy
Transmission and Analytical

INTRODUCTION

Al-Mg alloys are well-suited for marine applications due to their low density, ease of fabrication, structural durability, and most notably resistance to corrosion. Microstructural modification to the standard AA5083 through alloying and heat treatment can yield greater mechanical properties yet at the same time could increase corrosion resistance through the modification of grain boundary phases which are known to be beneficial for intergranular corrosion resistance.

TECHNICAL CHALLENGES

Traditional methods for preparing thin foils for TEM analysis often involves electropolishing with solutions consisting of methanol and nitric acid. This presents a problem since the same acid is the key ingredient utilized in standard intergranular corrosion testing which attacks susceptible grain boundaries.

Valid scientific identification and chemical analysis of the grain boundary constituents thus poses a challenge because phases may be fully or partially dissolved during specimen preparation.

OBJECTIVE

The objective of this research is to develop a TEM sample preparation technique that would not damage nor dissolve grain boundary phases so that characterization can assuredly be conducted and to compare this newly developed method with traditional techniques.

TEM SAMPLE PREPARATION METHODOLOGY

This new specimen preparation technique uses phosphoric acid to reveal grain boundaries (GB) that contain corrosive phases which are then selectively extracted using a Focus Ion Beam (FIB) instrument. If deleterious phases were to exist, the boundaries would be etched, while boundaries that are free of such phases are not. Regions of these attacked boundaries are then extracted by focused ion beam milling.

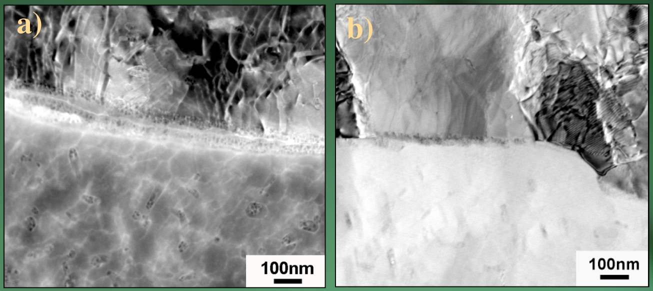

FIG. 1. STEM image showing a grain boundary with non-detectable phases likely dissolved by traditional electropolishing preparation methods.

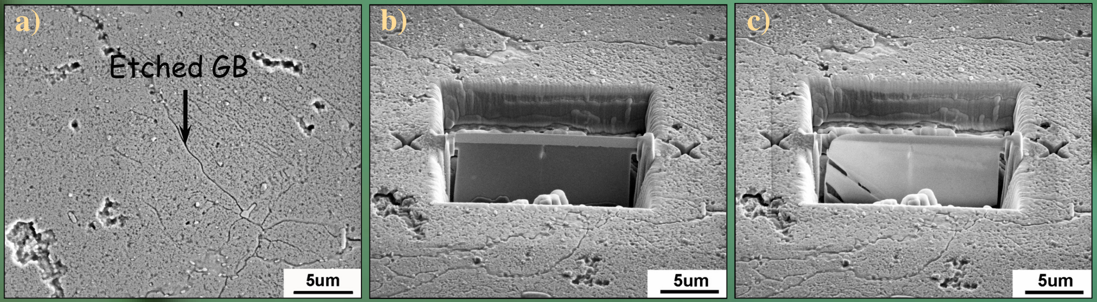

FIG. 2. Merged etching/FIB milling TEM sample preparation method: **a)** after applying phosphoric etching to reveal grain boundaries containing corrosive phases **b)** site specific focus ion beam milling and **c)** final thinning.

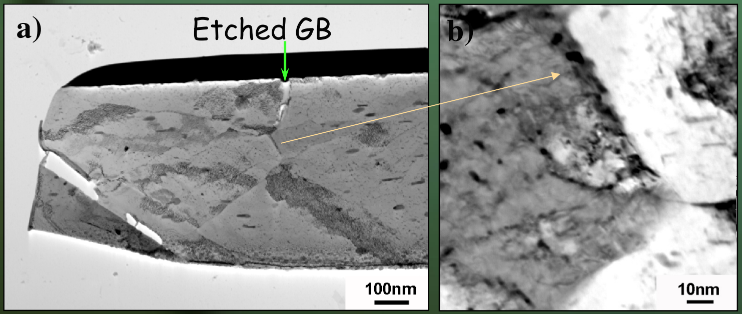

FIG. 3. a) Extracted TEM foil with partially etched grain boundary and **b)** underlying region with precipitates that are detectable and can be characterized through STEM imaging and analytical electron microscopy techniques.

EXPERIMENTAL DETAILS

A *Modified AA5083* was used to demonstrate the difference in TEM sample preparation techniques to identify grain boundary phases.

1. *Conventional Method:* A prethinned 3mm disc was electropolished using an electrolyte containing 75% Nitric Acid and 25% Methanol at -32° C/10V. After examination in the TEM, this technique results in GB phase dissolution. **(Figure 1)**

2. *New Method:* FIB milling in conjunction with 40% H_3PO_4 surface etching. A specimen was prepared using standard metallographic techniques with a final polish using a colloidal silica suspension. The sample was immersed in H_3PO_4 for 3 minutes at 35° C. An etched GB was then located and a sample extracted using a FIB. **(Figure 2 a-c)**

STEM imaging of the FIB prepared sample reveals GB phases which were later identified to be a modified τ-phase $Mg_{49}(Zn,Al)_{32}$ which is corrosive relative to the matrix. **(Figure 3 a-b)**

TECHNICAL SIGNIFICANCE

Focus ion beam milling of a phosphoric etched Al-Mg sample has proven to be a fast and reliable method to prepare TEM foils for alloys that contain grain boundary phases.

Through this method, it is now possible to properly correlate and identify grain boundary phases to the macroscopic intergranular corrosion behavior.