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ROLE OF INTERMETALLIC PARTICLES IN LOCALIZED CORROSION OF AA7075-T651

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ABSTRACT

The behavior of Al-Zn alloys such as AA7075-T651 is of particular interest owing to their potential applications in new designs due to high strength and good fatigue behavior. These high strength alloys are susceptible to localized corrosion in electrolytes associated with galvanic interaction between the matrix and various precipitated phases, including nm/sub- μm precipitates and large (10- μm scale) intermetallic particles (IMPs). The localized corrosion of AA7075-T6518, and the associated behavior of major IMPs, were studied using Scanning Kelvin Probe Force Microscopy, Scanning Electron Microscopy, Transmission Electron Microscopy, Energy Dispersive Spectroscopy, and electrochemical measurements. In particular, topography and Volta potential maps of IMPs and the matrix were collected using SKPFM to determine the distribution and relative nobility before and after exposure to sodium chloride solution. These combined measurements provide insight into the electrochemical behavior of IMPs and their effects on localized corrosion of AA7075-T651.

INTRODUCTION

The 7xxx Al-Zn alloys such as the alloy AA7075-T651 are designed to offer low density, high modulus, high specific strength, and superior fatigue performance advantages over conventional aluminum alloys for aerospace applications [1]. However, these alloys are susceptible to localized corrosion due to their heterogeneous microstructure [2]. As with other precipitation hardening aluminum alloys, localized attack of Al-Zn alloys in corrosive environments is associated with galvanic interactions between the matrix and various intermetallic phases, including various hardening precipitates, dispersoids and constituent particles [3, 4, 5], among which large (submicron to 10-micron scale) intermetallic particles (IMPs) are of particular importance as they have large volume fraction and are present in the matrix in large numbers [5, 6]. This raises concerns regarding failure of these alloys during service by localized corrosion and environmental cracking.

The micro/nano structure and electrochemical properties of IMPs and the associated localized corrosion in Al alloys have been extensively studied using a variety of techniques, including Scanning Kelvin Probe Force Microscopy (SKPFM) [3, 4], Scanning Electron Microscopy (SEM) analysis [3, 5], Energy Dispersive Spectroscopy (EDS) [5, 7], and Transmission Electron Microscopy (TEM) [4, 7, 8], and microcell measurements [6]. Studies have focused on a number of topics including the behavior of different types of IMPs [3, 4, 5, 6], pitting initiation along the IMP/matrix interface [4, 7, 9], electrochemical dissolution in different environments [6, 10, 11], kinetics of oxygen reduction reaction on surface of different IMPs [11], Volta potential change after electrolyte exposure [3, 5], and mechanism(s) of noble element (e.g. Cu) redistribution [12, 13]. These studies have provided considerable insight into the mechanisms of localized corrosion in the study of legacy 7xxx aluminum alloys.

In the present research, the IMP properties in AA7075-T651 and localized corrosion associated with them are studied using these methods. A series of different IMPs were identified and their chemistry and nanostructure were evaluated using different microscopy techniques and electrochemical measurements. Topography and Volta potential mapping was carried out using SKPFM to determine the distribution and relative nobility of each phase type (matrix and various IMPs) after polishing and after sequential exposure to sodium chloride solution. Post exposure features of IMPs were also reexamined using various microscopy techniques to determine the specific roles of the various IMPs. These combined results allow a better understanding of the electrochemical behavior of IMPs, galvanic coupling between IMPs and the matrix, and preferential attack to active contents in IMPs and matrix, and feedback the improved design and application of such new alloys.

EXPERIMENTAL PROCEDURE

Alloy samples of dimensions 7 mm × 7 mm × 3 mm were cut from AA7075-T651 alloy sheets, ground to 1200 grit using SiC paper with ethanol as lubricant, polished with 0.25 μm diamond paste in ethanol, and finally rinsed with ethanol. No ultrasonic cleaning was implemented to avoid IMP removal from the surface. Diamond-shaped micro-indenters were made on the sample surface to aid in the repeated location of regions of interest before and after exposure to electrolyte. In the exposure test, the samples were immersed into 0.01 M and 0.6 M NaCl aqueous solution and periodically removed, rinsed with water, air dried, and characterized using different techniques.

Topography and Volta potential mapping of samples before and after exposure to electrolyte were collected in air using SKPFM on a Veeco MultiMode 8 with Nanoscope V. The SKPFM tip was a Bruker OSCM-PT-R3 coated with titanium-platinum on front side and with aluminum on back side. The scan frequency was set at 0.1 Hz with 1024 pixels per scanning line. The tip was lifted 100 nm from the surface for the lift mode potential scan. All images were post-analyzed using NanoScope Analysis 1.5.

The microstructure and composition of selected regions were examined by SEM/TEM coupled with EDS/EELS. Representative regions containing IMPs after electrolyte exposure were cross sectioned by a Ga⁺ ion source in a FEI Helios Nanolab 600 Dual Beam Focused Ion Beam (FIB) for analysis in the depth of samples. The accelerating voltage was between 15-25 kV for SEM imaging and 30 kV for cross sectioning.

PRELIMINARY RESULTS

Three major types of IMPs in AA7075-T651 of general composition Al-Fe-Cu-(Mn), Mg-Si, and Al-Fe-Si-(Cu, Mn) were identified by SEM/EDS. There was an exact correlation between the spots of high or low Volta potential relative to matrix and the IMPs observed in the SEM image. Specifically, two types of large cathodic particles are generally in the size of 10-μm scale and stick out from the surface. The Al-Fe-Cu-(Mn) IMP was observed to be cathodic and to have a heterogeneous structure and chemistry by mapping of the topography and potential. A cluster of smaller IMPs of the same kind were also identified on both maps and may indicate the breakdown of a large IMP during cold work or even possibly during sample polishing due to the brittle nature of intermetallic phases. The cathodic IMP Al-Fe-Si-(Cu, Mn), however, had a very well defined edge along the IMP/matrix interface, indicating less effect of pitting initiation and galvanic dissolution in the interfacial region. The IMP Mg-Si is anodic to the matrix, and slightly lower on topography than the cathodic IMPs but still higher than the matrix. However, certain particles of this kind are even depressed relative to the general sample surface due probably to their preferential dissolution during polishing process. The chemical composition of these IMPs were also examined more precisely by TEM/EDS equipped in TEM. One of the Al-Fe-Cu-(Mn) IMPs exhibited a composition of Al₂Cu(Fe, Mn).

The same region was re-examined using SKPFM and EDS after 4-hour exposure to 0.1 M NaCl. Trenching developed in/around the Al-Fe-Cu-(Mn) IMP, which is expected due to the potential difference and heterogeneous structure. The trenching should be initiated by the preferential dissolution of Al and Mg in both the matrix and IMP because of their relative activity and the various structural defects in/around IMP. This IMP and its surrounding are enriched in Fe and Cu, and covered by a layer of oxide as suggested by EDS mapping of oxygen. Corrosion attack around the Al-Fe-Si-(Cu, Mn) IMP is not evident as no corrosion features were observed in this region, the edge of this IMP was still intact, and no apparent changes were found in the potential contrast. However, the anodic Mg-Si IMP was depressed significantly by 300-350 nm, and as a result, the oxide layer covered this region and made the surface Volta potential noble relative to nearby matrix as observed in the potential map.

Among the three major types of IMPs, Al-Fe-Cu-(Mn) played a dominant role in driving the galvanic dissolution of the matrix, Al-Fe-Si-(Cu, Mn) had a trivial effect on the matrix, and Mg-Si dissolved itself rather than causing attack in the matrix. However, both the dissolution of matrix and the self-dissolution of IMPs cause stress

accumulation in the matrix that could eventually lead to environmental cracking of the alloy and are worthy of further investigation. Even the electrochemical behavior of Al-Fe-Si-(Cu, Mn), and associated effects on localized attack to the matrix may be different in different aqueous environments, and thus additional study is needed.

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