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A microscopic study on the corrosion fatigue of ultra-fine grained and conventional Al–Mg alloy

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ABSTRACT

The corrosion behavior of a nanocrystalline (NC)/ultrafine grained (UFG) Al–Mg based alloy was investigated and compared to its conventional counterpart 5083(H111). The corrosion fatigue (CF) was studied with respect to pit initiation, pit location and crack propagation as a function of environment. Scanning electron microscopy (SEM) with EDS was used to analyze the fracture surface of the failed specimen with respect to pitting characteristics, crack propagation and corrosion product. Load vs. cycles to failure was measured and *S*/*N* curves were generated for the UFG Al–Mg based alloy and the conventional counterpart 5083 in air and seawater.

1. Introduction

Aluminum alloys are known for being ductile, lightweight and having excellent strength to weight ratios. Unfortunately, the ultimate strength of these alloys is not as high as conventional structural materials like steel. New advances in manufacturing techniques allow precise control over the microstructure evolution which permits tailoring of the microstructure, thus improving the tensile strength and other essential properties such as wear and fatigue. Al 5083 is a predominately aluminum–magnesium based alloy that is often used in marine and naval applications and could benefit from improved strength and fatigue resistance. Reducing the grain size of a material has been shown to improve properties of materials [1,2]. Cryomilling powders to produce nanocrystalline particles and subsequently consolidating into billet form followed by plastic deforming, is one way to produce materials with nanocrystalline (NC) or ultrafine (UFG) grains. UFG and NC materials from Al–Mg alloys have yielded tensile strengths between 520 and 740 MPa which is more than twice the ultimate tensile strength of polycrystalline Al 5083 materials (269–290 MPa) [1,2]. In addition to the high strength, high temperature and increased fatigue properties reported for these fine grained materials, corrosion properties are of particular interest for naval applications [3,4]. The corrosion behavior of fine grained aluminum and magnesium alloys is a significant concern because previous research has shown corrosion resistance to improve and deteriorate based on the processing method, composition, distribution of second phase particles and service environment [2,5–15]. Liao et al. [14] found that fine grained Mg–Al alloys exposed to 0.1 M NaCl solution had superior corrosion resistance when compared to micro-grained hot-extruded alloys of the same composition. They attributed the enhanced corrosion performance to the enhanced passivity of the oxide film that generated on the surface [14]. Similarly, Sharma and Ziemian [2] found the performance of NC/UFG Al 7.5 Mg alloys in atmospheric environment to depend significantly on pitting kinetics which varied based the processing conditions and chemical composition. Understanding the behavior of fine grained materials that will be used in structures, and that are subjected to loading (static and cyclic) while exposed to aggressive environments is of significant importance if they are to be viable for naval applications. The resulting combined action of loading and aggressive environment often results in stress corrosion cracking and corrosion fatigue; both ensuing in damage that significantly reduces a material's fracture resistance. Since reduced fracture resistance can result in serious or catastrophic failure, a study of the environmental degradation mechanisms and fatigue properties of fine grained materials is clearly needed if they are to be used in these applications.

Pitting corrosion is a known damage mechanism affecting the integrity of aluminum materials that are used in structures for naval applications. Most aluminum alloys contain a thin oxide layer on the metal surface which helps to reduce the corrosion rate. However, when localized breakdown of this film occurs, pitting corrosion is the result, thereby promoting accelerated dissolution of the underlying metal. Corrosion pits generally initiate due to a

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chemical or physical heterogeneity at the surface, such as inclusions, second phase particles, flaws, mechanical damage, or dislocations [2–4,16]. The composition of the material, amount, size and distribution of second phase particles and type of electrolyte will all have an effect on the corrosive mechanisms that result [17–19]. Burstein et al. [20] studied the origins of pitting corrosion, and revealed that pit nucleation occurs at the microscopic level with some metals displaying preferential sites for pit nucleation. Naval alloys contain various constituent particles for strengthening which play a significant role in corrosion pit formation [17–19]. Many studies have utilized optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM) techniques to better understand particle-induced pitting corrosion in aluminum alloys [2,16,21]. For the most part, corrosion fatigue initiates with pitting and subsequent crack formation, and ends with the propagation of the crack initiated at the base of the pits. As a result, it can be said that pitting directly activates crack initiation earlier [16–19] and can significantly affect the fatigue life of aluminum alloys used in naval applications.

The resistance to fatigue crack initiation and propagation of metals and alloys is also known to be influenced significantly by grain size [22]. Based on the Hall–Petch relationship, and the basis of experimental results obtained in conventional microcrystalline metals, it is widely accepted that an increase in grain size generally results in a reduction in the fatigue endurance limit. However, for nanocrystalline materials, this law to a certain extent is not observed, giving way to the so-called inverse Hall–Petch effect; which is a mechanism currently not well understood. Such lack of understanding is primarily a consequence of the lack of experimental data on the fatigue response of metals with very fine grains. Tensile properties and fatigue crack growth have been investigated for ultrafine grained (UFG) and nanocrystalline Al–Mg alloys [23–25]. However, a direct comparison of the cause of fatigue crack initiation, propagation and behavior in marine environments of the very small grain size Al–Mg alloy to larger grain sized materials of similar composition has not been made. Therefore, the objective of this study is to investigate the corrosion fatigue behavior of an UFG Al–Mg alloy and compare the results to a conventionally processed micrograined alloy of similar composition.

2. Experimental procedure

Two aluminum alloys were used to study the effects of a finer grain size and manufacturing method on the corrosion fatigue behavior of the aluminum–magnesium alloys. The materials chosen were a conventionally processed 5083-H111 Al–Mg alloy and an ultra fine grained (UFG) partially nanocrystalline (NC) Al–7.5 Mg alloy. The aluminum alloy 5083-H111 can be classified as a wrought alloy product. Al 5083-H111 was chosen because of its common marine applications and excellent corrosion resistance. Currently, the Navy uses Al 5083 for all topside marine atmospheric exposure conditions. The H111 temper was chosen based on the similarity to the manufacturing process of the UFG alloy. The compositions of the Al 5083-H111 and UGF Al–Mg alloys, in weight percent, are given in Table 1.

Table 1
Composition of Al–Mg alloys investigated.

Element	Alloy compositions in weight percent	
	5083-H111	UFG Al–7.5 Mg
Mg	4.90	7.50
Fe	0.31	0.09
Cr	0.13	–
Mn	0.40	–

2.1. Material processing

The UFG Al–7.5 Mg alloy was manufactured by the Boeing Company in collaboration with the University of California, Irvine. The material was processed through a combination of cryomilling and hot isostatic pressing. Spray atomized powders, with an average particle size less than 150 μm, were mechanically milled in liquid nitrogen through cryomilling to reduce the grain size. After cryomilling, the powder had a grain size of approximately 30 nm. The powder was then heated to 300 °C under a vacuum of 10^{-6} Torr for degassing. Powder was consolidated by hot isostatic pressing (HIP) at 250 °C under a pressure of 200 MPa. Finally, the material was extruded at an extrusion ratio of 20:1 for the UFG Al–7.5 Mg. In order to help interpret the data obtained in this study, reference is made to previous work investigating the localized corrosion and pitting behavior of these same alloys [2,26].

2.2. Specimen design

The shape and dimensions of test samples are shown in Fig. 1. The samples used in fatigue testing were smooth round dog-bone specimens, machined in the longitudinal direction, where the loading axis paralleled the extrusion direction. To investigate the fatigue behavior of the alloys, a smooth groove was machined into the samples to reduce the smooth gauge section and thus increase the foregoing area ratio. Effectively, the diameter of the specimen was reduced from the original 3.81 mm to nominally 1.6 mm at the notch. The notch radius was 6.35 mm. This was done to execute a timely test and because pre-tests without the smooth groove indicated the samples to be notch sensitive when they failed in the threaded grip section. Replicate samples were prepared from the same extrusion for each respective material. Round bars were first wire EDM'd (Electro-Discharge Machined) from the extrusion and then machined to the final dimensions. The surface finish of the material in the gauge section was polished to 1200 grit.

2.3. Fatigue testing

Uniaxial tension–tension fatigue tests were conducted at the LaQue Center for Corrosion Technology (LCCT) in Wrightsville Beach, North Carolina. The tests were executed at a stress ratio R (min/max) of 0.1 using a sinusoidal cyclic frequency of 20 Hz in air and 1 Hz in natural seawater. To generate S–N curves in air and seawater for the UFG Al–7.5 Mg alloy, fatigue specimens were loaded to various maximum stress levels between 173 and 420 MPa and the cycles to failure noted. For the conventional 5083 alloy, maximum stress levels between 173 and 310 MPa were used. Preliminary testing helped identify the best range of maximum stress values to use for each respective alloy. To test

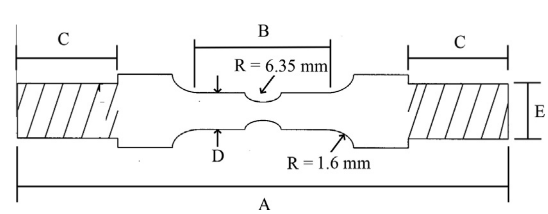

Fig. 1. Shape and dimensions of test specimen: smooth round tensile bar specimen with a round groove machined in the gauge section for corrosion fatigue testing. The dimensions are as follows: A = 50 mm, B = 12.7 mm, C = 3.8 mm, D = 12.7, E = 6.35 mm.