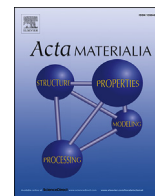




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Full length article

## Tensile stress and plastic deformation in aluminum induced by aqueous corrosion

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### ABSTRACT

Measurement of near-surface stress generated by metallic corrosion can reveal defects and material property changes relevant to structural degradation by stress corrosion cracking. In this study, stress and topography evolution were characterized during alkaline corrosion of high-purity aluminum. In situ stress measurements revealed tensile increases of the force per width (stress integrated over depth) that scale directly with the sample yield stress. Spectral analysis of the uniform dimpled surface pattern produced by corrosion showed that transient changes of its characteristic wavelength track closely with the force per width. Together, the stress and topography measurements imply that corrosion creates a plastically deformed metal layer. Tensile stress is attributed to the lattice contraction associated with metal vacancies introduced during dissolution. In agreement with this hypothesis, a vacancy diffusion model successfully predicted the time dependence and magnitude of the force response, as well as the observed scaling relation between plastic layer thickness and pattern wavelength. The driving force for vacancy formation is thought to arise from high hydrogen and low aluminum chemical potentials near the corroding surface, the latter imposed by the high dissolution potential relative to the equilibrium potential of aluminum.

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### 1. Introduction

Corrosion processes in combination with external or residual stress can induce embrittlement of structural metals, leading to premature and unexpected failure by stress corrosion cracking (SCC). While SCC has been studied intensively for several decades because of its wide economic significance, critical chemical interactions responsible for corrosion-enhanced degradation have not been fully clarified. In some systems such as high-strength 7xxx series Al alloys, corrosion is accompanied by absorption of hydrogen produced by cathodic water reduction, and SCC may be closely related to hydrogen embrittlement (HE) [1]. SCC and HE of Al alloys has been explained in terms of mechanisms such as hydrogen-enhanced local plasticity (HELP), hydrogen-enhanced decohesion (HEDE) or brittle hydride fracture [2,3].

Aside from hydrogen defects, several authors suggest that metal vacancies produced during corrosion may influence SCC and HE of various alloys. Vacancies are thought to promote degradation by

facilitating dislocation emission or climb at crack tips [4–6], decohesion [7], or void formation [8–10]. Formation of vacancies has been explained by direct injection into the metal by dissolution [4–6,11], tensile strain or cold work [9,10]. Also, strong binding of vacancies to absorbed hydrogen can result in dramatically enhanced concentrations of both vacancies and hydrogen near corroding interfaces, potentially assisting failure by HELP and HEDE mechanisms [12–14]. On the other hand, it is thought that vacancy injection during dissolution is kinetically challenged by the appreciable energy barrier to form lattice vacancies, so that high electrochemical potentials at the dissolving surface may be required [15]. Recent advances in atomistic simulations have led to the development of quantitatively predictive models for SCC and HE that include relevant length scales ranging from crack dimensions down to those of chemical interactions between atoms at crack tips [16–19]. While these models can simulate the detailed effects of defects on embrittlement, there remains a critical need to understand the metal-environment interactions during corrosion that control near-surface concentrations of defects participating in SCC.

In this study, we characterize formation of near-surface defects

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using in situ measurements of stress changes during corrosion. Hydrogen absorption and vacancy injection respectively expand and contract the metal lattice close to the surface, generating biaxial near-surface stress that in turn induces changes of sample curvature. High-resolution curvature measurements permit sensitive detection of near-surface hydrogen and vacancies during corrosion [20,21]. Chu and co-workers, using ex situ stress measurements, already found evidence that corrosion-induced tensile stress correlates with SCC susceptibility [22,23]. The present work focuses on defect generation during uniform alkaline corrosion of large-grain high-purity Al. Such samples avoid complications arising from localized corrosion processes at grain boundaries and second-phase particles. Alkaline corrosion of Al is accompanied by rapid hydrogen absorption [14,24], as well as formation of large numbers of subsurface nanoscale voids, the latter which may be attributable to condensation of near-surface vacancies produced during dissolution [25–27].

The present experiments employ a new technique, phase-shifting curvature interferometry, with significantly higher curvature resolution than traditional cantilever deflection methods [28,29]. The enhanced curvature resolution permits stress measurements in relatively thick sheet and foil samples, in contrast to thin metal films typically used for deflection techniques. Thicker samples enable study of material property effects in experiments with extended corrosion exposures. The present measurements revealed large tensile stress increases that scale directly with the sample yield strength. Spectral analysis of the dimpled topography produced by dissolution demonstrated that the characteristic wavelength of this uniform surface pattern scales with corrosion-induced stress, apparently the first indication of corrosion-induced plastic deformation. Model calculations show that both the thickness of the plastically deformed layer and the pattern length scale are determined by the diffusion penetration of vacancies injected by dissolution. The electrochemical driving force for vacancy diffusion-induced tensile stress and plasticity is explained in terms of the high hydrogen chemical potential and low aluminum chemical potential imposed respectively by the metal dissolution and hydrogen absorption processes.

## 2. Experimental methods

Aluminum samples were fabricated from either 1 mm thick sheet of 99.998% purity (Alfa Aesar), or 25  $\mu\text{m}$  thick foil of 99.999% purity (Alfa Aesar). Electron backscatter diffraction revealed a typical grain size of 200–400  $\mu\text{m}$  in the sheet material. Some of the sheet samples were cold-worked to 0.65 mm thickness using a hand mill. Foils were annealed at 550  $^{\circ}\text{C}$  for 52 h, increasing their grain size to the order of 100  $\mu\text{m}$ . Sample yield stress was estimated from the measured Vickers hardness (Wilson Tukon Hardness Tester) [30]. Usually the aluminum surface was not treated before dissolution. In one experiment, the aluminum sheet was electro-polished for 5 min at 30 V in a solution of 20% perchloric acid in ethanol. Alkaline corrosion of the Al samples was at open circuit in aqueous sodium hydroxide solutions. Topographic changes resulting from dissolution were characterized by field-emission SEM (FEI Quanta 250) and atomic force microscopy (AFM, Veeco Metrology Dimension 3100).

Detailed descriptions of the phase shifting curvature interferometry system and procedures used in stress measurements are provided elsewhere [29]. For stress measurements, both sheet and foil samples were cut into rectangular shape (3.5  $\times$  2.5 cm). Only one side of the sample was exposed to the corrosive solution, while curvature changes were determined by interfering light beams reflected on the opposite side. Thus, curvature measurements are not affected by corrosion-induced surface roughening. To produce

reflective surfaces for interferometry, gold films were transferred to one side of sheet samples using a template-stripping technique. Foils were bonded with epoxy to silicon wafers, which reflected the light beam and also functioned as elastically stiff substrates. Curvature and force per unit width changes are related by the thin-film Stoney approximation,  $\Delta F_w = [E_s h_s^2 / 6(1 - \nu_s)] \Delta \kappa$ , where  $\Delta F_w$  and  $\Delta \kappa$  are the force per width and curvature change; and  $h_s$ ,  $E_s$  and  $\nu_s$  are the thickness, elastic modulus and Poisson's ratio of the Al sheet or Si wafer.  $\Delta F_w$  represents the biaxial in-plane stress  $\sigma_{xx}$  integrated through the sample thickness, relative to that before corrosion, with the  $x$ -direction parallel to the Al surface.

## 3. Results and discussion

### 3.1. Stress measurements

All force measurements revealed significant tensile stress increases during alkaline corrosion. Fig. 1 compares stress evolution of Al sheet samples in 0.1, 0.5 and 1.0 M NaOH. Force per width in the figure represents the biaxial in-plane stress ( $\sigma_{xx}$ , with  $x$ -direction parallel to surface) integrated through the near-surface stress-affected layer, relative to that before corrosion. In the 1.0 M solution, the force increases initially in the compressive direction to a minimum value of to  $-3$  N/m at 0.8 min, after which tensile force begins to accumulate. Force change in the 0.1 and 0.5 M solutions is tensile from the beginning of dissolution. In each solution, tensile force increases for 7–17 min until a plateau is reached. The plateau force increases significantly with concentration, but the rate of force increase before the plateau is similar in all three solutions. The rates of both metal dissolution and accompanying hydrogen absorption increase significantly with pH over the range in Fig. 1 [24], suggesting that the stress response is controlled directly by the corrosion process.

Since Al has a moderate yield stress on the order of 100 MPa, tensile stress generated during corrosion could induce near-surface plastic deformation of aluminum metal. Fig. 2 shows the force per width transient in 1 M NaOH for three samples with close to 99.998% purity but different yield stress: annealed 25  $\mu\text{m}$  foil, 1 mm as-received sheet and the same sheet after cold rolling to 650  $\mu\text{m}$  thickness. The respective yield strengths of the annealed foil, as-received sheet and rolled sheet were estimated as 20, 90 and 120 MPa using Vickers hardness measurements. The force transient of the annealed foil but not the rolled sheet exhibits a compressive minimum of  $-3$  N/m at 1 min, similar to that of the as-received sheet. Both the average rate of tensile force increase and the plateau force increase significantly with yield stress. Indeed, the

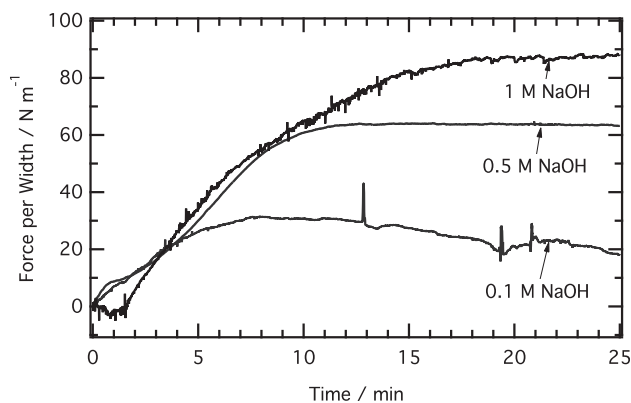


Fig. 1. Effect of NaOH concentration on force per width evolution during open-circuit alkaline etching.

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