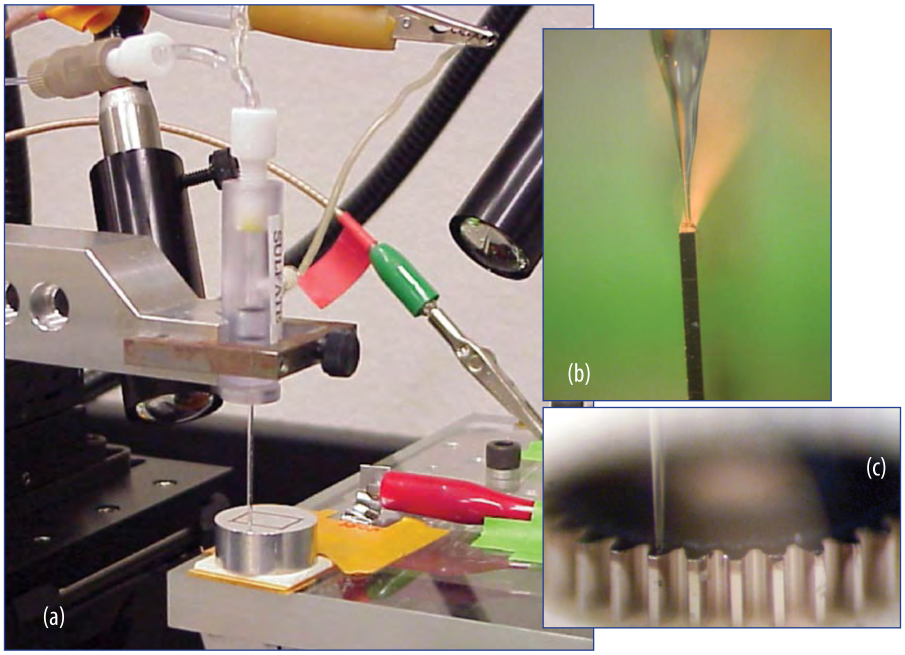
Discovery at the Interface of Science and Engineering: Science Matters!

Materials Science and Technology
Corrosion Science



Sandia technologies combine to detail pathways to corrosion

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Figure 1. (Above) (a) Microcapillary corrosion test system used to measure picoampere level currents at small length scales. (b) Assessing corrosion behavior electromachined Ni alloy. (c) Assessing local behavior on MEMS gear tooth.

Metallic corrosion impacts a range of materials applications from micro-electro-mechanical and microelectronic structures to engineered barriers for storage of high level radioactive waste. Although the size scale of these systems differs by orders of magnitude, a common feature of the metals (e.g., aluminum, titanium, tantalum, and nickel) used in their construction is the reliance on a thin, adherent metal oxide to impart passivity and yield long-term dimensional or structural integrity.

Corrosion scientists have developed theoretical models to describe how localized corrosion of the protective oxide barrier initiates, but until recently have not been able to directly validate the models due to a lack of access to the appropriate length scales (e.g., 1-10 nanometers). Using a unique combination of electrochemical atomic force microscopy, capillary-based electrochemistry, and state-of-the-art transmission electron microscopy, researchers at Sandia National Laboratories have been able to induce and characterize the nanoscale structure that emerges prior to loss of passivity. The clustering of cation vacancies in the metal oxide to form voids that then transition to pore structures has been successfully imaged, leading the way for studies of the critical changes necessary to initiate localized corrosion.

The developed approaches to electrochemical programming of local structure and subsequent characterization have also paved the way for validation of models that previously were not accessible in an experimental context. The emerging fundamental understanding is a necessary cornerstone for predicting reliability based on mechanistic behavior rather than empirical relationships.

For more information:

Technical Contact:
Kevin Zavadil, Ph.D.
505-845-8442
krzavad@sandia.gov

Science Matters Contact:
Wendy Cieslak, Ph.D.
505-844-8633
wrciesl@sandia.gov

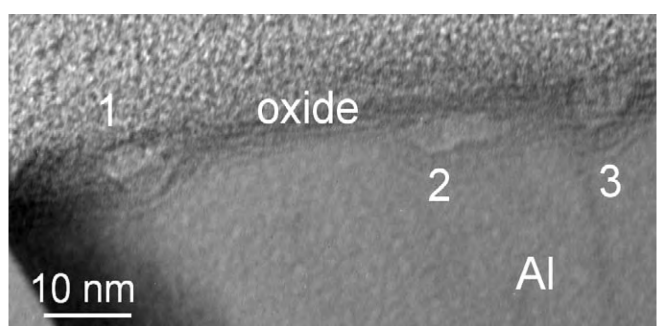

Figure 2. Transmission electron micrograph showing induced nanoscale morphology changes prior to passivity loss in aluminum: void in metal oxide (1), void at metal/oxide interface (2), pore in metal oxide that evolved from a void (3).

Sandia National Laboratories

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND 2006-5313P

NNSA National Nuclear Security Administration

The primary goal of Sandia National Laboratories' corrosion program is to prevent or mitigate any undesirable effects that this form of metal degradation can have on high-reliability electrical and structural components. The diversity and breadth of our technical expertise and our focus to obtain science-based engineering solutions to corrosion problems and concerns make our program unique. Because of its multidisciplinary nature, the corrosion staff closely interacts with researchers from many of Sandia's technology centers (e.g., materials science, nanoscience, computational science, and geochemistry). Sandia has significant expertise in four corrosion-related areas: atmospheric corrosion, aqueous corrosion, electrochemical science, and failure analysis.

Atmospheric Corrosion

In addition to mechanistic information, our experimental activities provide the ability to perform device-level assessments. State-of-the-art, mixed flowing gas systems produce temperature and humidity controlled environments containing ppb levels of contaminants. We can perform accelerated testing in standardized conditions. We have extensive experience with both copper and aluminum corrosion, related primarily to corrosion in electronics.

Aqueous Corrosion

Because aluminum alloys are the dominant structural material used in Sandia-designed hardware, most of our aqueous-related expertise centers around characterizing and controlling its localized corrosion behavior. Conventional electrochemical techniques are routinely used, and we are proficient in applying and evaluating most coating technologies, including chromate conversion, anodized, and other organic coatings. Recently, we have gained considerable expertise applying microelectrode-based techniques to understand localized corrosion phenomena.

Electrochemical Science and Sensors

Corrosion mechanisms and electrochemical processes are addressed with both traditional and nontraditional techniques. Interfacial and surface chemistry is studied using electron and ion spectroscopies in an ultrahigh vacuum environment and with complementary analyses in a contiguous electrochemical chamber. A non-traditional approach being pursued involves corrosion-sensing schemes integrated directly into material systems of interest. For example, this technology is useful for studying atmospheric corrosion, where reactions take place in ultrathin, localized, condensed-water layers on metal surfaces.

Failure Analysis

Sandia has many years of experience in performing corrosion-related failure analyses of mechanical and electrical components. High-quality optical microscopy, scanning electron microscopy/energy-dispersive spectroscopy, electron microprobe, and other analytical facilities complement our materials understanding and experience. We combine our corrosion knowledge and experience with that of other materials-science disciplines (ceramics, polymers, etc.) to determine the cause of failure. We provide root-cause analysis, remediation, and prevention information to both internal and external customers.

SELECTED ACCOMPLISHMENTS

- Applying our evolving analytical tool set to assess the effects of corrosion on the reliability of several electronic devices.
- Developing and applying several microelectrode-based techniques to determine pit-initiation mechanisms in aluminum.
- Developing a nondestructive technique to determine the quality of field-aged, chromate-conversion coated aluminum.
- Designing and fabricating an in-situ sensor technology to characterize spatially localized surface conditions (chemistry and water phase).
- Providing understanding and solutions to several corrosion-induced failures that have occurred in industrial production environments.

PUBLICATIONS

K.R. Zavadil, J.A Ohlhausen, P.G. Kotula, "Nanoscale Void Nucleation and Growth in the Passive Oxide on Aluminum as a Prepitting Process," *J. Electrochem. Soc.* **153** (8) B296-B303 (2006).

K.R. Zavadil, P.G. Kotula and J.A. Ohlhausen, "Interfacial Structure and Composition as Controlling Factors in Void Formation at the Passive Oxide – Aluminum Interface," ECS Trans. **1** (4) 139-149 (2006).

K.R. Zavadil, P.G. Kotula, and J.D. Chavez, "Interfacial Voids as Possible Precursor Sites for Pore Formation and Pitting in Aluminum," in Pits and Pores II: Formation, Properties and Significance for Advanced Materials, P. Schmuki, D.J. Lockwood, Y.H. Ogata, M. Seo and H. Isaacs, PV2004-19, p. 254-264, The Electrochemical Society Proceedings Series, Pennington, NJ (2005).

L.M. Serna, K.R. Zavadil, C.M. Johnson, F.D. Wall, J.C. Barbour, "A Critical Implanted Cl Concentration for Pit Initiation on Aluminum Thin Films," *J. Electrochem. Soc.* **153** (8) B289-B295 (2006).