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FUNDAMENTAL STUDIES OF PASSIVITY AND PASSIVITY BREAKDOWN

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ACHIEVEMENTS

This report summarizes the findings of our fundamental research program on passivity and passivity breakdown under Grant No. DE-FG03-84ER45164, "Fundamental Studies of Passivity and Passivity Breakdown." This grant was held from September 1, 1990 to October 31, 1991 by the Principal Investigator (Dr. D. D. Macdonald) while at SRI International. However, on July 1, 1991, Dr. Macdonald joined The Pennsylvania State University as Professor of Materials Science and Engineering and as Director of the Center for Advanced Materials. Subsequently, the grant was activated at Penn State University as DE-FG02-91ER45461.

During the past three and one half years in this program (including the three year incrementally-funded grant prior to the present grant), we developed and experimentally tested various physical models for the growth and breakdown of passive films on metal surfaces. These models belong to a general class termed "point defect models" (PDMs), in which the growth and breakdown of passive films are described in terms of the movement of anion and cation vacancies. Specifically, our work during the past three and one half years of this program resulted in:

(1) Derivation of a theory for the steady-state properties of passive films on metal surfaces. This theory was found to give an accurate description of the dependence of the steady-state film thickness and current on potential and pH for passive nickel in phosphate solutions, as derived in this work, and for a variety of metals and alloys in various aqueous media as determined from data from the literature.

(2) Demonstrated that the passive film on nickel is a cation conductor whereas that on tungsten is an oxygen ion conductor. Ionic conduction in the passive film on nickel involves the irreversible ejection of cations from the film into the solution at the film/solution interface.

(3) Development of deterministic models for the distributions in the critical voltage and induction time for passivity breakdown. The distributions in these parameters are attributed to distributions in the breakdown sites in terms of the cation vacancy diffusivity.

(4) Derivation of distribution functions for the critical voltage (V_c) and induction time (t_{ind}) for passivity breakdown using different distributions for the breakdown sites with respect to the cation vacancy diffusivity. The continuous distributions used include the log and normal distributions. Other distributions (student "t" and χ-square) are now being developed. Numerical analysis of the derived

distributions in V_c and t_{ind} is now being performed for comparison with experimental data.

(5) Development of a coupled environment model for pit growth. We are currently coupling this model with the distribution functions for V_c and t_{ind} to derive a deterministic treatment for predicting damage functions for pitting corrosion (distribution in pit depth versus exposure time).

(6) Development of the solute-vacancy interaction model to account for the effects of alloying elements on the critical voltage and induction time for pit nucleation on an alloy surface.

(7) Unification of the point defect and solute/vacancy interaction models to explore the effects of alloying elements on the distributions in the critical voltage (V_c) and induction time (t_{ind}) for passivity breakdown. The model was then used to account for critical pitting potential data for Fe-Cr-Ni-Mo alloys taken from the literature.

(8) Measurement of distributions in the critical breakdown voltage and induction time for pit nucleation on Ni-xAl, Ni-xTi, and Ni-xMo alloys in buffered boric acid and buffered chloride solutions. These data agree with the predictions of the solute-vacancy interaction model for the effects of alloying elements on passivity breakdown.

(9) Development of a "point defect" model for the segregation of alloying elements into passive films. The predictions of the model are in accord with experimental segregation data obtained using a new technique, SALI (*s*urface *a*nalysis by *l*aser *i*onization), on dilute Ni-xAl, Ni-xTi, and Ni-xMo ($x < 8$ wt%) alloys.

(10) Detection and explanation of photoinhibition of passivity breakdown as observed on nickel in buffered KCl solution. Photogeneration of electron/hole pairs apparently quenches the electric field in the passive film thereby reducing the driving force for the movement of cation vacancies from the film/solution interface to the metal/film interface. The lowered vacancy flux results in a positive shift in the breakdown voltage and hence in enhanced resistance to pitting.

(11) Extension of the Chao, Lin, and Macdonald theory for the impedance characteristics of passive films by incorporating kinetic effects caused by reactions involving vacancies at the metal/film and film/solution interfaces. We show that the impedance of passive nickel in phosphate buffer solutions (pH = 9-11) at high frequencies ($f > 100$ Hz) is dominated by the kinetics of reactions occurring at the metal/film interface, whereas the impedance at low frequencies ($f < 1$ Hz) is dominated by the movement of vacancies through the film.

(12) Development of methods for measuring photoelectrochemical impedance functions for metal- and semiconductor-solution interfaces by modulating the intensity of an incident, monochromatic light beam and by transforming photocurrent transients from the time domain to the frequency domain. By using an He/Ne laser with a photon energy of 1.96 eV (632 nm), we measured the photoelectrochemical

impedance spectra for n-Si and p-Si in contact with sodium hydroxide solution over wide ranges of modulation frequency and applied bias potential. We are currently developing theoretical models to explain the impedance data in terms of the photogeneration of electron/hole pairs and their recombination via surface states, and in terms of the reactions of electrons and holes with redox species in the solution.

(13) The use of Kramers-Kronig (K-K) transforms of electrochemical impedance spectroscopy (EIS) data to ascertain whether electrochemical systems satisfy the linearity, causality, and stability constraints imposed by linear system analysis. We examined how well experimental EIS data satisfy the K-K transforms by exploring one particular class of impedance functions known as constant phase impedances. We show unequivocally that constant phase impedances, which frequently include those for passive films at low frequencies, transform provided that the loci are restricted to the right side of the complex plane.

(14) Development of a mixed potential model (MPM) for calculating corrosion potentials in the heat transport circuits of water cooled nuclear reactors. To our knowledge, this is the first deterministic estimation of this important parameter, which is of fundamental importance in determining the susceptibilities of sensitized ausenitic stainless steels to intergranular stress corrosion cracking in nuclear reactors. We have used the MPM to derive corrosion potential maps for in-vessel components of two domestic boiling water reactors (BWRs) - Dresden-2 and Duane Arnold - and we used the maps to explore the efficacy of hydrogen water chemistry (additions of hydrogen to the feedwater) as a means of preventing fracture in operating plants.

(15) Development of a fully deterministic coupled environment fracture model (CEFM) for estimating crack growth rates in sensitized Type 304SS in water cooled nuclear reactor heat transport environments. To our knowledge, this is the only model for stress corrosion cracking that satisfies charge conservation explicitly, by stipulating that the current exiting the crack month must be consumed quantitatively by a net cathodic process occurring on the external surfaces. The CEFM is found to yield crack growth rates that are in excellent agreement with both laboratory and plant data and therefore appears to be well-suited as a basis for estimating operating lifetimes of plant components.

A central hypothesis of our theoretical work on the effect of alloying elements on passivity breakdown is that highly charged solutes, such as Mo^{6+} substituted into NiO passive films, form ion pairs with mobile cation vacancies of the type $Mo^{4\bullet}_{Ni}$ ..... $V^{2'}_{Ni}$. This interaction is described as a chemical equilibrium using the Debye-Huckel and Onsager-Fuoss ion-pairing theories, originally developed for aqueous electrolyte solutions but later used to describe vacancy-impurity interactions in semiconductors. This approach accounts very well for the effect of molybdenum on the pitting resistance of stainless steel and for

the effects of Al, Ti, and Mo on the breakdown characteristics of nickel in buffered chloride solutions. The most effective alloying elements are those that

(1) Segregate to the greatest extent into the passive film (Mo > Ti > Al).

(2) Possess the greatest charge relative to the host cation (i.e., $Mo_{Ni}^{4\bullet}$ $Ti_{Ni}^{2\bullet}$ > Al_{Ni}).

The extent of segregation was attributed principally to the high dielectric constant of the thin, electronically conducting passive film and hence to the ability of the film to "solvate" the substituted alloy solute. The magnitude of the charge is important because it determines the electrostatic energy of interaction with the negatively charged cation vacancies and hence the extent of ion-pair formation. However, these charges are also screened by oppositely charged entities (e.g., cation vacancies and electrons), and allowance for this effect is made using the Debye-Hückel screening radius.

As noted above, photoinhibition of passivity breakdown, as observed in this program for nickel in buffered chloride solutions, is a direct manifestation of coupling between the electronic and lattice defect structures of the films. Because the films are highly doped with vacancies, which act either as acceptors ($V_M^{\chi^x}$) or donors ($V_O^{\cdot\cdot}$, where χ is the oxide stoichiometry ($MO_{\chi/2}$), the model that emerges for the electronic structure of the film is that of a metal-n^+-i-p^+-solution junction, with the relative p^+ and n^+ characters depending on the relative concentrations of metal and oxygen vacancies, respectively.

As with classically degenerate n^+-i-p^+ junctions, the potential drop occurs entirely across the depletion region, with the electric field strength determined by the compensating processes of charge separation owing to isoenergetic tunneling from filled levels in the valence band to unfilled levels in the conduction band and electron/hole recombination, possibly via surface states. The photogeneration of electron/hole pairs quenches the electric field, thereby reducing the driving force for the transport of cation vacancies from the film/solution interface to the metal/film interface. Thus, incident light is expected to inhibit passivity breakdown, as observed experimentally. We are now exploring photoinhibition as a practical means of protecting equipment in the power generation industry from pitting corrosion and stress corrosion cracking. This concept is being explored under an EPRI Exploratory Research grant.

Our work on stress corrosion cracking was prompted by the considerable concern that is developing in the nuclear industry as to the susceptibilities of in-vessel components in the nation's boiling water reactors (BWRs) to stress corrosion cracking, particularly at

high neutron fluences. The development of the MPM and the CEFM provide, for the first time, deterministic methods for estimating corrosion potentials and crack growth rates for in-vessel components in BWRs, and hence could form the basis for an effective life prediction methodology.

FUTURE RESEARCH THRUSTS

The work carried out over the past several years in this program has resulted in the development of several new and unique methods and concepts for exploring corrosion processes. Of particular significance is the development of deterministic methods for describing the distribution functions for the nucleation of pits on metal surfaces, because only stoichastic (empirically statistical) methods had been employed in the past. The new method permits, for the first time, estimation of pitting corrosion damage functions for industrial systems. Indeed, we are using the theoretical foundation developed in this program to estimate pitting damage functions for gas-fired condensing heat exchangers for the Gas Research Institute. Additionally, the solute/vacancy interaction model developed in this program has led to a set of "rules" for designing new, corrosion resistant alloys that may be used to improve the performance of energy conversion, conservation, and production systems.

We believe that the key to developing a comprehensive understanding of the phenomenon of passivity is the characterization of the coupling between the electronic and (crystallographic) defect structures of the passive film. Possibly the most effective way of characterizing this coupling is to monitor, simultaneously, the responses of both structures to appropriate perturbations. Thus, we have employed a combination of electrochemical impedance spectroscopy (EIS), which excites the crystallographic defect structure, and photoelectrochemical impedance spectroscopy (PEIS), which excites the electronic defect structure. By measuring the impedances while varying the conjugate excitation function (i.e. by measuring the EIS function while varying the photon energy and flux and by measuring the PEIS function while varying the voltage), we hope to provide new insight into the remarkable properties of passive films. We hasten to add that while the experimental techniques (including those developed in the present program) are relatively mature, the theoretical methods and concepts required to interpret the data are still in their infancy. Accordingly, much of our effort will be devoted to developing more accurate theoretical techniques for interpreting impedance data and relating the data to the properties of passive films.

Finally, we have embarked on an effort in this program to develop a more deterministic description of stress corrosion cracking, with particular emphasis on the cracking of components in the heat transport circuits of water-cooled nuclear power

reactors. Because measurements of crack growth rate and corrosion potential are difficult (and, in many cases, impossible) to make inside reactor pressure vessels, we do not believe that satisfactory empirical models will be developed within the near future. Accordingly, in our view, the only viable approach is to develop fully deterministic treatments based on established physico-chemical laws (e.g., the conservation of charge). Appropriately, an effective component life estimation methodology will require the development of fully deterministic treatments for the nucleation of cracks from pits and flaws and for estimating crack growth rates; these treatments are provided by our work in this program on passivity breakdown and on developing the coupled environment fracture model for stress corrosion cracking in water cooled nuclear reactors.

PUBLICATIONS AND PRESENTATIONS

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37. D. D. Macdonald and M. Urquidi-Macdonald, "Electrochemical Modeling of the Cores of Light Water Reactors," presented at The 180th Electrochemical Society Fall Meeting, Phoenix, AZ, Oct. 13-18, 1991.

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41. D. D. Macdonald and M. Urquidi-Macdonald, "Progress Towards Modeling the Cores of Water-Cooled Nuclear Reactors," DOE Corrosion Contractors Meeting, Brookhaven National Laboratory, Sept. 19-20, 1991.

Awards

1. D. D. Macdonald, 1991 Carl Wagner Award, The Electrochemical Soc. - Award made at the 180th Society Meeting in Phoenix, AZ, Oct. 13-18, 1991.

2. D. D. Macdonald, 1992 Rodney Willis Whitney Award, The Nat. Assoc. Corros. Eng. - To be awarded at CORROSION/92, Nashville, TN, April 27 - May 1, 1992.

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