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Experimental Application of Design Principles
in Corrosion Research *

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ABSTRACT

Experimental design criteria for corrosion investigations are based on established principles for systems that have uniform, or nearly uniform, corrosive attack. Scale-up or scale-down may be accomplished by proper use of dimensionless groups that measure the relative importance of interfacial kinetics, solution conductivity, and mass transfer. These principles have been applied to different fields of corrosion which include materials selection testing and protection; and to a specific corrosion problem involving attack of a substrate through holes in a protective overplate.

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229

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Introduction

The behavior of materials in aggressive environments involves the complex interplay between thermodynamic, kinetic, and transport processes. The balance between competing influences is a dynamic one which responds to the appearance and disappearance of multiple phases such as films at interfaces, and changes as the rates of the several individual chemical reactions change with time. Before an attempt is made to describe in detail the complicated interactions of a corrosion system, one should describe the macroscopic behavior. This brings one naturally to the derivation and use of models to give a quantitative measure of the relative importance of different influences. Because models lead to quantitative predictions, one can then assess and dismiss incorrect assumptions about the system behavior, and can systematically investigate details of the performance of a material. The purpose of this paper is to describe the contribution that modeling may make to corrosion systems, especially in the areas of materials testing, selection and protection. Several examples of such applications will be described. The emphasis will be placed upon the experimental utility of principles of current and potential distributions in practical applications.

Because of the electrochemical nature of many corrosion processes, the techniques of modeling in electrochemical systems can be adopted. Such techniques have been applied with success for many years, so their use has proven to be effective. Most notable of these are the determination of current and potential distributions. This involves the mathematical analysis and solution of Laplace's equation for the electric field to obtain the current distribution on electrode surfaces. This is equivalent to determining the rate of the electrochemical reactions at the reaction interface.

The first attempt to model electrochemical systems involved the application of techniques developed for the mathematical physics of electricity and magnetism theory. The results demonstrated immediate usefulness in attempts to model electrodeposition and electroplating. In these processes, the pattern and distribution of the electrode reaction were of obvious importance, and remain so today. Battery systems have benefitted as well because the calculations have helped in the design of grids where there is uniform, complete utilization of battery materials. Modeling activities in these systems were at first limited to geometric arrangements where rigorous mathematical solutions could be obtained. More recently, approximation methods were adopted when the systems became too complicated to be treated rigorously, especially when the effects of diffusion were considered important. The development of numerical solution techniques by computers have given modelers a new tool, especially for the treatment of complex interactions that are inherent to corrosion systems. By applying these techniques, useful models that can assist the corrosion scientist and engineer have been produced. A brief description of some of the advances in modeling corrosion systems will be given, but not in a complete or comprehensive fashion. Some of the key principles of treating a corrosion system will be discussed first, so that some of the background and terminology may be developed. Particular applications will then be discussed, first in materials testing, and finally in materials selection.

Principles of Current and Potential Distribution Calculations

The prediction of current and potential distributions involve the solution of Laplace's equation for the potential. This is equivalent to specifying that charge is conserved, or that there are no sources or sinks for current in the body of the electrolyte. This is a classic equation which has been studied in great detail in many branches of mathematical physics. The equation in one dimension is

$$\frac{d^2\phi}{dx^2} = 0$$

and in three dimensions it is the partial differential equation

$$\frac{\partial^2\phi}{\partial x^2} + \frac{\partial^2\phi}{\partial y^2} + \frac{\partial^2\phi}{\partial z^2} = 0$$

The solution of this equation must be determined for appropriate boundary conditions. The boundary conditions on electrodes are of three general types: 1) a constant potential on the electrode surface; 2) a linear relationship between the derivative of the interfacial potential and the current; and 3) a nonlinear relationship between the potential derivative and the current at the surface.

A boundary condition of a constant potential along the electrode is appropriate when the electrode reaction(s) are very fast. In the literature, the solution to this classic problem is called the primary current or potential distribution. The solution has been obtained for some simple systems, and the results may be generally stated as follows:

- (1) The primary distribution does not involve the solution conductivity and is therefore independent of the identity of the solution,
- (2) The distribution depends on geometric ratios of the system, but not on an absolute length or size. By the principle of similarity, it may be concluded that for two similar systems to have identical behavior, the geometric ratios must be identical, but the absolute size of the system is not important.

Most corrosion systems are not described by this primary distribution, because interfacial electrode kinetics are important, even dominant, in deter-

mining the potential or current distribution. The effect of interfacial kinetics is introduced through boundary conditions. The potential and current distributions still depend on the same geometrical parameters as the primary distribution, but also on an additional parameter which characterizes the influence of ohmic resistance and electrode kinetics. Three mathematical expressions have been used to describe the current-voltage relationships expected of electrode kinetics. A linear relationship between current density and electrode overpotential, i.e.,

$$i = \frac{i_o(\alpha_a + \alpha_c)F}{RT} \eta_s$$

has been found to be appropriate when the kinetics are fairly fast or the overpotential is small. It is really the ratio of i/i_o which must be small for the linear boundary condition to be used. In this equation, α_a and α_c are kinetic parameters for the anodic and cathodic reactions respectively, i_o is the exchange current density and η_s is the overpotential of the electrode, i.e., $\eta_s = V - \phi_o$ and measures the potential difference between the electrode V , and the potential just outside the electrical double layer ϕ_o . Both potentials are measured with respect to a common reference electrode. The current density relationship is matched with the expression for the current in solution normal to the electrode surface, i.e.

$$i = \frac{i_o(\alpha_a + \alpha_c)F}{RT} \eta_s = -\kappa \left. \frac{\partial \phi}{\partial x} \right|_{x=0}$$

The distribution now depends on the dimensionless parameter (1),

$$J = \frac{i_o(\alpha_a + \alpha_c)F L}{\kappa RT}$$

where L is some characteristic dimension of the electrode. The value of J represents the relative importance of ohmic influences in solution as compared to the

interfacial kinetic influence. Large values of J indicate the dominance of ohmic effects, and the distribution approaches the primary distribution as $J \rightarrow \infty$. Small values of J indicate a greater kinetic influence. The general result of a greater kinetic influence is to make the current distribution more uniform on the surface, as compared to the primary distribution. Use of these general results allows one to predict behavior, as well as to provide a basis for scaling a system up or down, since similar systems will show similar behavior for the same values of the geometric ratios (from the primary distribution) and the same values of J . It should be noted that the behavior of the electrode material is described by $(\alpha_a + \alpha_c)$ and i_o , and the environment is characterized by the solution conductivity κ .

It has been found empirically that the electrode kinetic behavior may be given by the nonlinear Tafel relationship

$$i = i_o \exp \left(\frac{\alpha_a F}{RT} \eta_s \right)$$

(taken here as an anodic reaction), which is appropriate at high current densities, i.e., $i \gg i_o$. The parameter (1)

$$\delta = \frac{\alpha_a F L}{\kappa RT} i_{avg}$$

measures the relative magnitude of the ohmic influence to the kinetic influence. Large values of δ are characteristic of ohmic dominance, and the potential or current distribution approaches the primary distribution as δ increases.

A general nonlinear expression for the current density is the Butler-Volmer Relationship

$$i = i_o \left[\exp \left(\frac{\alpha_a F}{RT} \eta_s \right) - \exp \left(- \frac{\alpha_c F}{RT} \eta_s \right) \right]$$

which reduces to the linear form at low current density, and is approximated by the Tafel relationship at high current density. If neither approximation is appropriate, this general expression must be used and the parameters which describe the system are (1)

$$\frac{\alpha_a}{\alpha_c}, J, \text{ and } \frac{(\alpha_a + \alpha_c)FL}{\kappa RT} |i_{avg}|$$

The linear current-voltage relationship has been popular because the mathematics are more amenable to solution, and an analytic solution may be obtained for many systems. The Tafel relationship may be linearized about some average potential, so that a linear boundary condition is again obtained. This linearization has been useful in electrodeposition and other applications where the potential on the electrode surface does not drastically vary from the average potential. In corrosion applications linearization is often invalid because the size of the system is large enough that there exists a large potential variation over the surface. The use of computerized numerical finite difference techniques makes it possible to use the general boundary condition that will describe the current distribution at all current levels over the entire surface. Such solutions are more realistic and often more accurate than the results obtained with an approximate boundary condition.

The use of numerical finite difference methods have been used to obtain the results shown in Figure 1. The disk electrode is embedded in a coplanar, surrounding plane. The disk is anodic and the surrounding plane is either an insulator or a cathode. The cathodic plane will cause the current density to concentrate at the outer edge of the disk, as compared to the insulator case

($J_p = 0$). This models a galvanic corrosion situation which has been discussed in the literature (12). More will be said about this example later.

In summary, the following are the important general results of current and potential distribution modeling:

- (1) the primary distribution of current or potential is a function of geometric ratios and is independent of the chemistry of the system;
- (2) the effect of finite electrode kinetics is to produce a more uniform current distribution than the primary distribution;
- (3) the distribution of current is characterized by the dimensionless parameter J for linear kinetics, the parameter δ for Tafel kinetics, and the parameter $\left(\alpha_a/\alpha_c\right)$, J and $\frac{(\alpha_a + \alpha_c)FL}{\kappa RT} |i_{avg}|$ in general. The parameters J and δ measure the relative importance of ohmic and kinetic influences, and;
- (4) Systems with similar values of geometric ratios and polarization parameters demonstrate similar behavior.

Materials Testing

The current and potential distribution calculations described above may be directly applied to corrosion systems by substituting the corrosion current density, i_{corr} , for i_0 . It is also assumed that the corrosion potential is constant in time. Several examples of this type of application to corrosion testing have been investigated and are described in this section. The objective of this section is to suggest improvements and limitations on laboratory testing techniques to assure well defined results.

An important aspect of cathodic protection is to determine the size of the electrode material which may be maintained under protection for a certain supplied current. In the laboratory, it is difficult to conduct the testing of large pieces of material, therefore calibrations can help to scale the size from laboratory to field conditions. Smyrl and Newman (2) have identified the variation of potential that could be expected on a rotating disk electrode surface under diffusion control. It was found that the maximum difference in potential would be between the center and edge of the disk and the difference would be

$$\Delta\phi = \frac{0.363 r_o i_{avg}}{\kappa}$$

The maximum permissible potential variation ($\Delta\phi$) is sought which will maintain the disk under cathodic protection, but not waste current by overdriving the system to evolve hydrogen. This potential difference then is used with the solution conductivity (κ) and average current density (i_{avg}) to calculate the maximum size of the disk (r_o). A disk of larger radius will not be protected over a part of the surface, or will evolve excess hydrogen over some of the surface. Another example of this type of calculation in cathodic protection, but without diffusion, has been given by Newman (3) for a pipe whose interior is to be cathodically protected by placing a counter electrode at the end of the pipe. The result can be determined from the following equation,

$$L = \sqrt{r_o \kappa \Delta\phi / i_w}$$

where L is the length of the pipe that may be protected, r_o is the radius of the pipe, κ is the solution conductivity, i_w is the current that must be supplied to the wall to maintain protection. $\Delta\phi$ is the maximum potential difference that may

be allowed if excess hydrogen is to be avoided at one end and yet have the other end protected. These two examples can be extended to other situations and demonstrate that modeling can help to design experiments in cathodic protection testing.

Laboratory experiments to determine corrosion parameters should be designed so that reliable and well-defined measurements are made. One of the problems that may occur in making polarization measurements is that the current density distribution is nonuniform and therefore part of the surface supports more current than the rest. If the nonuniformity is large, the average current density may not yield good polarization parameters. Tiedemann, et al. (4) have calculated the error that may occur during kinetic measurements employing a disk electrode with nonuniform current distribution over the surface. These results may be applied to corrosion systems by substituting i_{corr} for i_o . The estimated error for i_{corr} could range from -50% to more than + 300% depending on the J parameter and the position of the reference electrode (4). The error is small for small values of J and is zero at $J = 0$, i.e., for high conductivity, small electrodes, and slow kinetics.

One may deal with the current distribution problem by using electrode geometries that have uniform primary distribution. The average current density will then be identical to the local current densities measured at any point on the surface. For this approach to be effective the measured currents must be below the diffusion limited current to eliminate any influence of diffusion. Several geometries have a uniform primary distribution which include: (1) two concentric spheres; (2) two concentric cylinders (neglecting end effects); (3) two disk electrodes which fill either end of a cylindrical tube; and (4) a hemisphere embedded in an insulating plane. The last geometric configuration is the easiest to construct and use. None of these geometries have been popular in corrosion testing, although there is sufficient justification for their use.

Methods have been devised to minimize the effect of ohmic nonuniformity when experimentally employing geometries other than those listed. In these cases the polarization parameters are extremely important. Specifically, the parameters, J and δ , provide a criterion for estimating the errors caused by nonuniformity, as discussed by Tiedemann, et al. (4), and suggest how one may minimize this effect. Generally, small values of J (or δ) ensure relatively uniform current densities. If the corrosion current and the electrical conductivity of the testing solution can be determined, the size of the electrode can then be varied to ensure a small value (approximately 10^{-2}) of J (or δ). If the corrosion current is neither known nor can be estimated from previous experimental data, one must determine whether the current density was uniform after the testing has been completed. This can be done by using the measured value of i_{corr} from the linear polarization method, and using the electrode size (e.g., the radius of a disk electrode), and solution conductivity to give a value of J . If the value of J is small, the measurement needs no current distribution correction, and if it is large, the measurement should be repeated using a smaller electrode. A similar approach, but now using the parameter δ , could be employed for Tafel polarization measurements.

To illustrate these points specific values for the J and δ parameters will be calculated. For a corrosion current of 0.1 mA/cm^2 , and the parameter values given below

$$R = 8.315 \text{ Joules/deg-mole}$$

$$T = 298.15 \text{ K}$$

$$F = 96,493.5 \text{ coulombs/equivalent}$$

$$\kappa = 4.12 \times 10^{-3} \text{ ohm}^{-1} \text{ cm}^{-1} \text{ (i.e., 0.01 M HCl)}$$

$$r_0 = 1 \text{ cm (radius of a disk electrode)}$$

$$(\alpha_a + \alpha_c) = 1$$

J was calculated to be

$$J = 0.945$$

A J value of this magnitude would require only a small correction for nonuniform current density. If the radius of the disk electrode were increased to 10 cm, the value of J would increase to 9.45 and therefore a sizeable correction would be required. On the other hand, for a corrosion current density of $1 \mu\text{A}/\text{cm}^2$, assuming the other parameters retained their same value, the value of J would be calculated to be 9.45×10^{-3} and 9.45×10^{-2} for a radius of 1 cm and 10 cm, respectively. In both cases the corrections would be negligible. The calculation for δ is similar except that the average current density is used instead of i_{corr} . For the following typical values

$$i_{\text{corr}} = 10^{-6} \text{ A}/\text{cm}^2$$

$$i_{\text{avg}} = 10^{-4} \text{ A}/\text{cm}^2 \text{ (i.e., } i_{\text{avg}} \gg i_{\text{corr}} \text{)}$$

$$\alpha_a = 0.5$$

and employing the same parameter values used above, the calculated values of δ would be

$$\delta = 0.472 \text{ (} r_o = 1 \text{ cm)}$$

$$\delta = 4.72 \text{ (} r_o = 10 \text{ cm)}$$

Even though the corrosion current density is small, the ohmic nonuniformity may be significant for the large disk. The technique for making the correction for nonuniform current distribution for Tafel polarization is somewhat complicated and difficult.

These calculations serve to emphasize that experimental variables may be manipulated to ensure that the J (or δ) values are small for the test measurements, and thus current distributions will be uniform. The average current density is then equal to the local current density.

To summarize this section, the description of some design parameters for cathodic protection of a disk and pipe were described. The size of the electrode that may be protected is related to the maximum potential which can be tolerated, the magnitude of the current density, and the value of the solution conductivity. For corrosion rate determination, the J and δ polarization parameters give a measure of the current distribution uniformity, and one may alter the experimental parameters to minimize them and make the current density uniform. The combination of small electrode size, high solution conductivity, and low corrosion current densities will make J (or δ) small to ensure a relatively uniform current distribution. This would then give reliable corrosion rate measurements.

Materials Selection

Once the testing phase has been completed, the selection of a material for a particular application is normally made on the basis of the lowest corrosion rate. It would seem that modeling could make no further contribution, beyond ensuring that the best corrosion rate data has been generated for use in making the selection. Modeling corrosion systems beyond this point would not be justified for those applications where the material would not be subjected to imposed currents, except for galvanic attack as will be discussed below. However the catastrophic failure of materials from crevice corrosion, pitting corrosion, and other forms of localized corrosion demonstrates that the long term behavior of a material cannot always be predicted from the normal corrosion testing discussed above. The laboratory tests might be extended to cover the normal service life, but this is usually not acceptable. Instead models can be effectively employed for predicting long term behavior. The models would help to reveal the dominant influences on the macroscopic behavior of a corrosion system, which would reveal causes and suggest remedies. The application of model calculations would also reveal diagnostic

behavior in certain areas that could be observed during testing and evaluation to aid in the selection of materials that would be resistant to localized attack. These two benefits of modeling, i.e., revealing long-term behavior of a real system, and revealing diagnostic testing criteria, will be discussed to show their applicability in several examples.

The modeling of galvanic corrosion systems has been discussed in the literature. If the J (or δ) parameters for both the cathode and anode are small, the current density on both electrodes will be uniform. The current density on each electrode depends on the potential driving force and the relative areas of the anode and cathode. Stern (5) first looked at this system, and Oldham and Mansfeld (6) have investigated it more recently. For larger values of J (or δ) the ohmic effects became important and cause nonuniform currents on the surfaces. Waber and coworkers (7) published a series of articles on galvanic corrosion which included ohmic effects for linear polarization. Subsequent work (8-11) on circular geometries has also used linear polarization boundary conditions. Recently, the results of using more general boundary conditions have been published (12) for the anodic disk embedded in a cathodic surrounding plane. The results in Figure 1 show the nonuniform distribution of current expected on the disk when ohmic effects are important. The influence of coupling the disk to the surrounding plane is to make the nonuniform distribution even more nonuniform near the disk-plane boundary. The boundary attack may be enhanced by increasing the polarization parameters (J_p or δ_d). This can be accomplished by decreasing the solution conductivity of the electrolyte. In situations where the disk is cathodic, and the surrounding plane is anodic, the solution conductivity could be increased to spread the attack over the entire anode surface and decrease the localized attack at the interface. Values for J or δ would likewise decrease. Waber (7) and others (8-11) have found

these general results for rectangular systems as well and have discussed the effect of decreasing the solution thickness over the galvanic boundary. As the thickness decreases, the attack is concentrated at the boundary, similar to the effect of reducing the solution conductivity.

Modifying the distribution of the galvanic attack can have important consequences, as dramatically demonstrated by the corrosion of composites (13). In this paper, the galvanic corrosion of a boron fiber-aluminum alloy composite was investigated. The bulk of the boron fiber was inert in the electrolytic solution but a narrow annulus of a boron-aluminum intermetallic around the fiber was cathodically active. This intermetallic was produced during high temperature fabrication, and galvanically caused the corrosion of the surrounding aluminum matrix. The J value of the aluminum matrix was high and caused the attack to be concentrated near the fiber-matrix boundary. The result of a long term attack like this would be loss of adhesion at the interface and significant losses in mechanical strength.

Modeling may also help to formulate materials selection criteria that would be helpful in rejecting material which will be susceptible to long-term localized attack. Such an example has been discussed by Alkire, et al. (14) for differential aeration. In brief, the finding was that a material which has a polarization curve such as that shown in Figure 2 is susceptible to differential aeration attack. A large cathode which supports the reduction of dissolved oxygen will accelerate the attack on oxygen-starved anodic regions. The diagnostic criteria necessary to observe differential aeration on the tested material is the presence of an oxygen cathodic current in the passive region. Materials which show this current-voltage behavior will be susceptible to nonuniform attack caused by nonuniform oxygen supply to the surface. This diagnostic criteria was generalized and applied to a

materials selection program for containers for an ocean service environment (15). Several materials were tested and were not found to have the diagnostic behavior for differential aeration. None of them have been reported to have this localized attack in other investigations.

Summary

The principles of current or potential distribution calculations were described. The nonuniformities expected for ohmic control are known to be moderated by interfacial electrode kinetics. The polarization parameters which characterize the relative importance of ohmic and kinetic effects were shown to have important applications in materials testing. Examples of design parameters for cathodic protection were discussed. Applications of current and potential distribution calculations in materials selection were found to be important in describing long term behavior, for example in galvanic corrosion. Such calculations also reveal diagnostic criteria which may be used to reject materials which would be susceptible to localized attack such as differential aeration.

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FIGURE 1.

CURRENT DISTRIBUTION ON A DISK IN A CONDUCTING PLANE
FOR DIFFERENT VALUES OF THE PLANE POLARIZATION PARAMETER, J_p .

FIGURE 2 POLARIZATION CURVES FOR AN ACTIVE-PASSIVE METAL IN SOLUTIONS WITH OXYGEN AND WITHOUT OXYGEN