

Mechanism of Action of Corrosion-Inhibited Highway Deicers

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The corrosion rates of steel exposed to salt solutions inhibited with several phosphate or phosphonate salts together with salts of magnesium, calcium, or zinc were measured to determine their effectiveness as low-corrosion deicers. At levels present in commercially available deicers, ortho- and polyphosphates mixed with magnesium or zinc salts provided strong inhibition of chloride corrosion. Linear polarization resistance (LPR) was used to monitor the corrosion rate of an orthophosphate-inhibited deicer over time, showing that the corrosion rate remained very stable and low in the presence of the inhibited deicer. The overall decreases in the corrosion rate of the inhibited salt relative to those of plain salt determined by LPR and weight loss were 89 and 81 percent, respectively, indicating good agreement between the two methods. Electron microscopy and energy dispersive spectroscopy were used to analyze steel surfaces exposed to different deicer formulas. Surface studies indicate that the inhibitors become incorporated into the surface of the steel, causing the formation of a compact, uniform layer that may reduce the corrosion by forming a barrier between the metal and the corrosive environment.

Historically, salt has been the deicer of choice for roads, highways, and bridges and will probably continue to be so because of its high ice-melting capacity, efficient ice-melting characteristics, and low cost. However, the low cost of salt is mitigated by the fact that it results in corrosion damage to motor vehicles, bridges, and the highway infrastructure. Estimates of the cost of deicer corrosion damage to vehicles and infrastructure range from \$2 billion to \$4.5 billion per year (1). To address this problem there is currently a great deal of interest in the development of alternative, low-corrosion deicing products.

One approach to developing low-corrosion deicing products has been to use the salts of organic acids such as calcium magnesium acetate (2). Since these organic deicers do not contain the aggressive chloride ion they are relatively less corrosive than salt. However, in some cases these organic compounds do not have the high deicing effectiveness of salt, and they may be prohibitively expensive (3). Another approach to developing low-corrosion deicers has been to add small amounts of chemical corrosion inhibitors to deicing salt. The corrosion inhibitors are designed to dissolve in the deicing brine melt and limit the corrosion caused by exposure to the runoff. To choose the most appropriate inhibitor formulations for deicing mixtures it is necessary to understand the mechanism of action of inhibitors in the deicing brine environment. This paper presents the results of recent investigations of the mechanism of action of corrosion-inhibited deicers on exposed carbon steel surfaces.

CORROSION RATE MEASUREMENT

Corrosion rates were measured on 3 percent deicer solutions. Concentrations of 3 percent were chosen to provide the most aggressive concentration for testing inhibitor effectiveness. Uhlig (4) points out that the corrosiveness of salt solutions to steel reaches a maximum at 3 percent and then begins to fall off because of decreasing oxygen solubility. The 3 percent concentration is also observed in deicing brine runoff concentrations measured in the field (5). Corrosion measurements were made on coupons of 1010 carbon steel because this is fairly representative of the steels in automobiles, highway fixtures, and bridge components.

Phosphorus-based inhibitors were chosen for the present study because these are used in a number of commercial anticorrosive deicing products. The formulas tested in the study were taken from commercially available corrosion-inhibited deicing products. All of the deicers tested were salt based, consisting of sodium chloride mixed with a phosphorus-containing inhibitor and a divalent metal salt. Formulas containing three different classes of phosphorus-based inhibitors were chosen: orthophosphates, polyphosphates, and organic phosphonates.

Orthophosphates, polyphosphates, and phosphonates are good candidates as deicer corrosion inhibitors. They are relatively inexpensive and nontoxic, and they are well known to be effective inhibitors in such industrial applications as cooling water treatment (6), potable water treatment (7), protection of steel in seawater (8), and protection of automobile cooling systems (9). Previous studies exploring the potential of polyphosphates as inhibitors for seawater indicate that the effectiveness of polyphosphates in salt solution can be increased by the addition of calcium, magnesium, or zinc salts (8,10). Thus, magnesium, calcium, and zinc salts may be present in the commercial phosphate-phosphonate-inhibited deicers to increase the inhibition effectiveness. The relative importance of the phosphate-phosphonate and divalent metal ion in inhibiting the corrosion of deicing salt remains to be determined. This information is necessary to determine the optimum amount of each inhibitor for an effective low-corrosion deicer.

Corrosion rates were measured on all test formulas by weight loss measurements consistent with the guidelines of ASTM G31 (11). One formula, the orthophosphate composition, was chosen for simultaneous measurement of the corrosion rate by weight loss and linear polarization resistance (LPR) to determine if LPR could be used to quickly and accurately monitor the corrosion rates of steel in deicing solutions. LPR is a commonly used electrochemical technique that measures instantaneous corrosion rates and that has been shown to correlate well with weight loss measurements for a wide variety of metals and corrosive media (12), including concrete-embedded rebar (13), although no measurements of the corrosion

rate of concrete-encased rebar were made in the present study. The technique involves imposing a small potential change on a metal sample. The slope of the resulting potential-versus-current curve at the corrosion potential is termed the polarization resistance (R_p). The actual corrosion rate can be calculated from the polarization resistance by the Stern-Geary equation (12):

$$i_{\text{corr}} = (\beta_a \beta_c) / 2.3(\beta_a + \beta_c) \cdot 1/R_p \quad (1)$$

where i_{corr} is the corrosion current (corrosion rate), and β_a and β_c are the anodic and cathodic Tafel slopes, respectively. The Tafel slopes are determined by taking the slope of the log i -versus- E polarization curves in the Tafel region, that is, the region where the curve is linear. Polarization scans were run on independent samples to determine the Tafel constants, but acceptable Tafel regions were not obtained. Cathodic polarization curves began to slope steeply within 30 mV of the corrosion potential, possibly due to oxygen reduction or passive film breakdown, which distorts the Tafel behavior in this range. Similar behavior has been observed for steel exposed to solutions of calcium magnesium acetate (14). Consequently, values of β_a and β_c of 110 mV/decade were used to calculate corrosion rates. These values have been used by other investigators to calculate corrosion rates when the actual Tafel constants were not known (14).

SURFACE ANALYSIS

A variety of surface studies have been done on steel exposed to phosphate-type inhibitors in different environments. Electron diffraction studies indicate that steel exposed to solutions of dihydrogen phosphate form a surface film that is made up of $\gamma\text{-Fe}_2\text{O}_3$ and $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ (15). Steel inhibited by calcium and sodium hexametaphosphate in salt solution has been shown to form a viscous surface film with a composition corresponding to a complex hydrated compound of iron, calcium, sodium, and metaphosphate (8). Electron spectroscopy for chemical analysis (ESCA) studies on a mild steel tube from a pilot plant cooling tower of a stabilized phosphate corrosion inhibition program indicated the formation of a surface layer that contained carbon, phosphorous, calcium, iron, and oxygen (16).

To gain information about the mechanism by which the inhibited deicers work and determine if surface behavior similar to that seen in other environments can be observed, scanning electron microscopy (SEM) was used to obtain photographs of steel surfaces exposed to salt and various inhibited deicers. In addition to information on the changes in surface morphology caused by the inhibited deicers provided by electron microscopy, further information was gained by analyzing the elemental compositions of the surfaces by using energy dispersive spectroscopy (EDS). Examined together, the physical appearance of the film that forms on a metal surface, the elements detected in the surface film, and the corrosion rate of the metal provide basic information about the corrosion process of a metal exposed to a given deicer.

PROCEDURE AND MATERIALS

Weight Loss Measurement

Deicers containing phosphate and polyphosphate inhibitors were obtained directly from their commercial sources. Samples of the phosphonate-inhibited deicer were obtained from the stockpile of

an end user. Four coupons of 1010 steel of $2.54 \times 5.08 \times 0.16$ cm ($1 \times 2 \times 0.0625$ in.) were immersed in 3 kg of each deicer solution. All concentrations of the deicer solutions were 3 percent and were made with deionized water (18 M Ω). The coupons were totally immersed in the solutions except for two 1-hr drying periods a day, 5 days per week, when they were suspended in air. The solutions were replaced with freshly made 3 percent solutions of the same type once a week. After 4 weeks of exposure weight loss measurements were made according to ASTM G31. The percent protection was calculated by Equation 2 (weight loss measurements were also made on uncorroded coupons exposed to the same cleaning procedure as the test coupons; the average weight loss due to cleaning was subtracted from the weight loss of the test coupons before calculating percent protection):

$$\text{Percent protection} = 100 \cdot (W_s - W_d) / (W_s - W_w) \quad (2)$$

where

- W_s = weight loss of the salt control,
- W_d = weight loss of the inhibited deicer sample, and
- W_w = weight loss of the water control sample.

Thus, percent protection calculated in this manner provides a measurement of the inhibition of salt-induced corrosion since the calculation subtracts the amount of corrosion arising from pure water. A value of greater than 100 percent protection indicates a corrosion rate less than that observed in pure water, and a value of less than 0 percent indicates a corrosion rate greater than that observed with plain salt.

LPR Measurement

Simultaneous LPR and weight loss measurements were made by the same procedure described above except that the coupons were prepared in the manner described for electrode specimens in SHRP Standard H-205.7 (17). After preweighing a 22-gauge stainless steel wire, the wire was attached to each coupon with a brass screw and nut through a hole drilled in the top. The top portions of the coupons were coated with liquid electrical tape, including the connection to the steel wire, so that 3.0 cm (1.18 in.) of the coupon was left exposed on the bottom. Four sets of four coupons each were exposed to solutions of salt and inhibited salt. Polarization resistance measurements were made consistent with the method outlined in ASTM G59 (18) by using a Schlumberger SI 1286 electrochemical interface with software written by Capcis March Ltd. A graphite rod obtained from the Carbide/Graphite Group, Inc., was used as the counter electrode. The reference electrode was a saturated calomel electrode (SCE) coupled to the solution via a Luggin probe. Four sets of four coupons each were tested for salt and the inhibited deicer. Each set of coupons was immersed in 3 kg of solution and was given one 1-hr drying period per day, 5 days per week. (Coupons in the LPR experiment were given only one drying period per day for increased measurement convenience. Upon completing the LPR measurements on a given day, the coupons were given their 1-hr drying period and were then allowed to remain in solution overnight to ensure that the corrosion potential and rate had restabilized before taking readings on the following morning.) Trapezoidal integration of the corrosion currents measured over 4 weeks was done to calculate the average corrosion currents for the salt and inhibited deicer solutions, i_s and i_d , respectively. At the end of the 4 weeks of exposure, the coupons were cleaned and weight loss mea-

TABLE 1 Corrosion rates of 1010 Steel in 3 Percent Deicer Solutions

Deicer / Inhibitor	MPY ^a	% Protection
Salt / No Inhibitor	13.3 (1.5)	0
Water control	3.2 (0.4)	100
Salt / Phosphonate + CaCl ₂	15.0 (0.8)	-17
Salt / Hexametaphosphate + ZnSO ₄	-1.6 (0.02)	115
Salt / Tripolyphosphate + MgCl ₂ ·6H ₂ O	0.3 (0.04)	128
Salt / Orthophosphate + MgSO ₄	0.6 (0.1)	125

^a Standard deviations are listed parenthetically.

measurements were made in the same manner described earlier. Because of the difficulties in measuring electrochemical corrosion rates in the absence of electrolyte, a water control was not run in this experiment, and percent protections could not be calculated as described earlier to permit comparison of the electrochemical and weight loss measurements. Therefore, comparison of the two methods was made by calculating the percent inhibition simply by using salt as the standard by the equation

$$\text{Percent inhibition} = 100 \cdot (i_s - i_d)/i_s \text{ or } (W_s - W_d)/W_s \quad (3)$$

where w refers to weight loss values and i refers to corrosion currents as defined earlier.

SEM and Energy Dispersive Surface Analysis

Coupons of 1010 steel [2.54 × 5.08 × 0.16 cm (1 × 2 × 0.0625 in.)] were exposed to 3 percent solutions of inhibited deicer and salt in the same manner that was used to make the weight loss corrosion measurements. Coupons were exposed for both 1 and 4 weeks. After exposure was complete the coupons were rinsed with deionized water, rinsed with methanol, and allowed to air dry. The

coupons were subsequently vapor deposited with carbon, and the measurements were made by SEM and EDS. Electron microscopy was performed with a JEOL 840II scanning electron microscope, EDS was performed with a Tracor Northern TN-5500. All samples were photographed at a magnification of ×250.

RESULTS AND DISCUSSION OF RESULTS

Corrosion Rate Measurements

The data in Table 1 show the results of weight loss corrosion rate measurements on the test deicer formulas. The data indicate that orthophosphate and polyphosphate formulas all gave corrosion rates lower than those observed in deionized water. The amount of inhibitor in the phosphonate-containing deicer was fairly low (less than 0.1 percent). The corrosion rate observed for this product probably indicates that higher levels of the phosphonate inhibitor are required to achieve a high level of corrosion inhibition.

LPR was used to monitor the corrosion rate of coupons exposed to the orthophosphate deicer formula and a salt control as a function of time. Figure 1 shows the changes in corrosion potential and

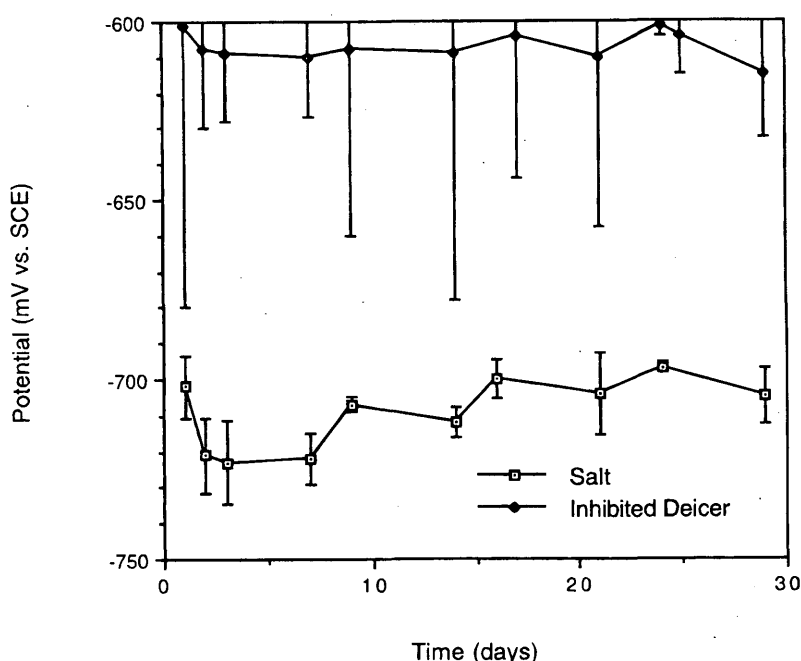


FIGURE 1 Corrosion potential of 1010 steel coupons exposed to 3 percent deicer solutions (error bars indicate 95 percent confidence intervals).

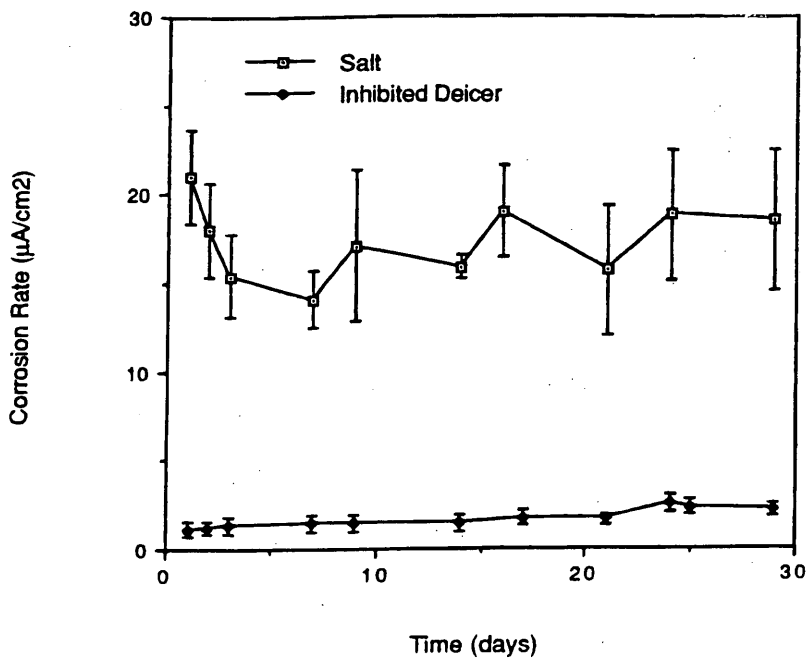


FIGURE 2 Corrosion rate of 1010 steel coupons exposed to 3 percent deicer solutions (error bars indicate 95 percent confidence intervals).

Figure 2 shows the corresponding instantaneous corrosion rates of these steel samples. The data given in Figures 1 and 2 are only for the salt solutions with and without inhibitor; electrochemical measurements in deionized water were not made because of the difficulty of making electrochemical measurements in the absence of electrolyte. The corrosion potential provides an indication of the relative degree of passivation of a metal surface. Shifts to more negative corrosion potential are a qualitative indication of active corrosion (19). Figure 1 shows that the inhibited deicer causes the steel potential to shift by more than 100 mV to less corrosion active potentials. The potential of steel exposed to inhibited deicer is much more variable than that of steel exposed to the salt solution (indicated by the much larger error bars in Figure 1), but even within experimental error there is a consistent, significant shift to more positive potentials, suggesting that the inhibited deicer helps preserve passivation in the presence of the aggressive chloride ion.

The corrosion potential only provides a qualitative indication of whether the steel is likely to corrode or not. For an unambiguous determination of corrosion it is necessary to measure actual corrosion rates. This can be seen for salt and the inhibited deicer in Figure 2, which provides quantitative confirmation of the effect suggested by Figure 1. It can be seen that within 1 day the corrosion rate in the inhibited solution has already reached a minimum, and the corrosion rate does not change significantly over 30 days of exposure.

To determine the accuracy of the LPR technique in monitoring deicer corrosion over time the average corrosion rate of the samples was measured by weight loss at the end of the experiment and was compared with that calculated by a trapezoidal integration of the instantaneous corrosion rates depicted in Figure 2. Since electrochemical corrosion rate measurements were not attempted for the water control, the percent protection could not be calculated in the same manner as that used to calculate the values in Table 1. However, it is possible to compare the percent decrease in the corrosion

TABLE 2 Corrosion Rates of 1010 Steel Measured by LPR and Weight Loss

	Plain Salt	Salt + Orthophosphate/MgSO ₄
Weight Loss Corrosion Rate (MPY)	5.3	0.99
LPR Corrosion Current (µA/cm ²)	473	51
Weight Loss % Inhibition	-	81%
LPR % Inhibition	-	89%

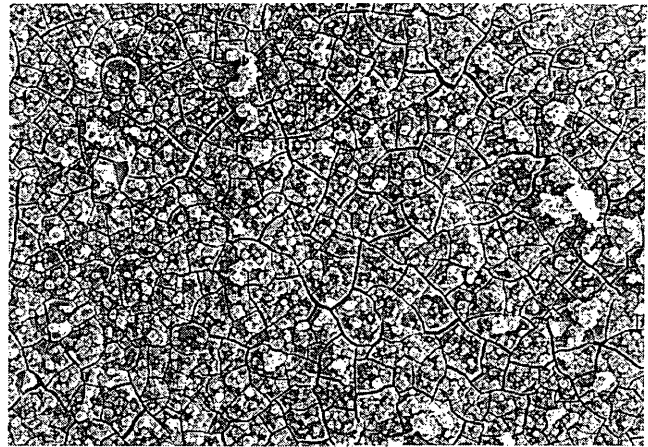
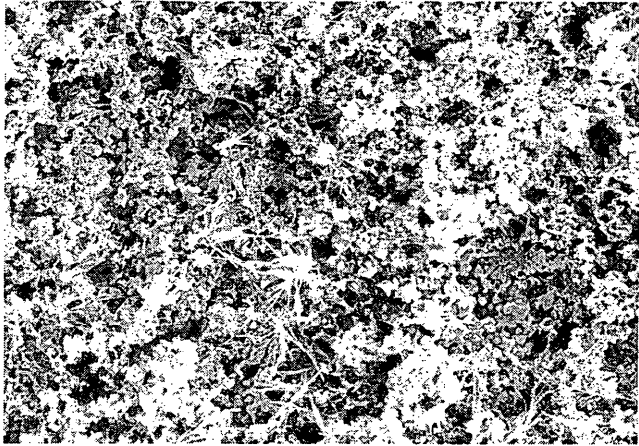


FIGURE 3 Scanning electron micrograph of steel exposed to 3 percent salt solution for 1 week.

FIGURE 4 Scanning electron micrograph of steel exposed for 1 week to a 3 percent solution of salt inhibited with zinc and hexametaphosphate.

rate of the inhibited salt relative to that of the plain salt control from the weight loss and electrochemical measurements. The corrosion rates and percent inhibition calculated by weight loss and LPR are given in Table 2. The relative corrosion rates measured by the two techniques agree very well. Weight loss measurements indicate an 81 percent reduction of the corrosion rate of the

inhibited deicer relative to that of salt, whereas corrosion rates measured by LPR indicate an 89 percent reduction in the corrosion rate relative to that of salt. Thus, LPR provides an accurate, rapid means of measuring deicer corrosion rates and permits observation of changes in the corrosiveness of the deicer over time.

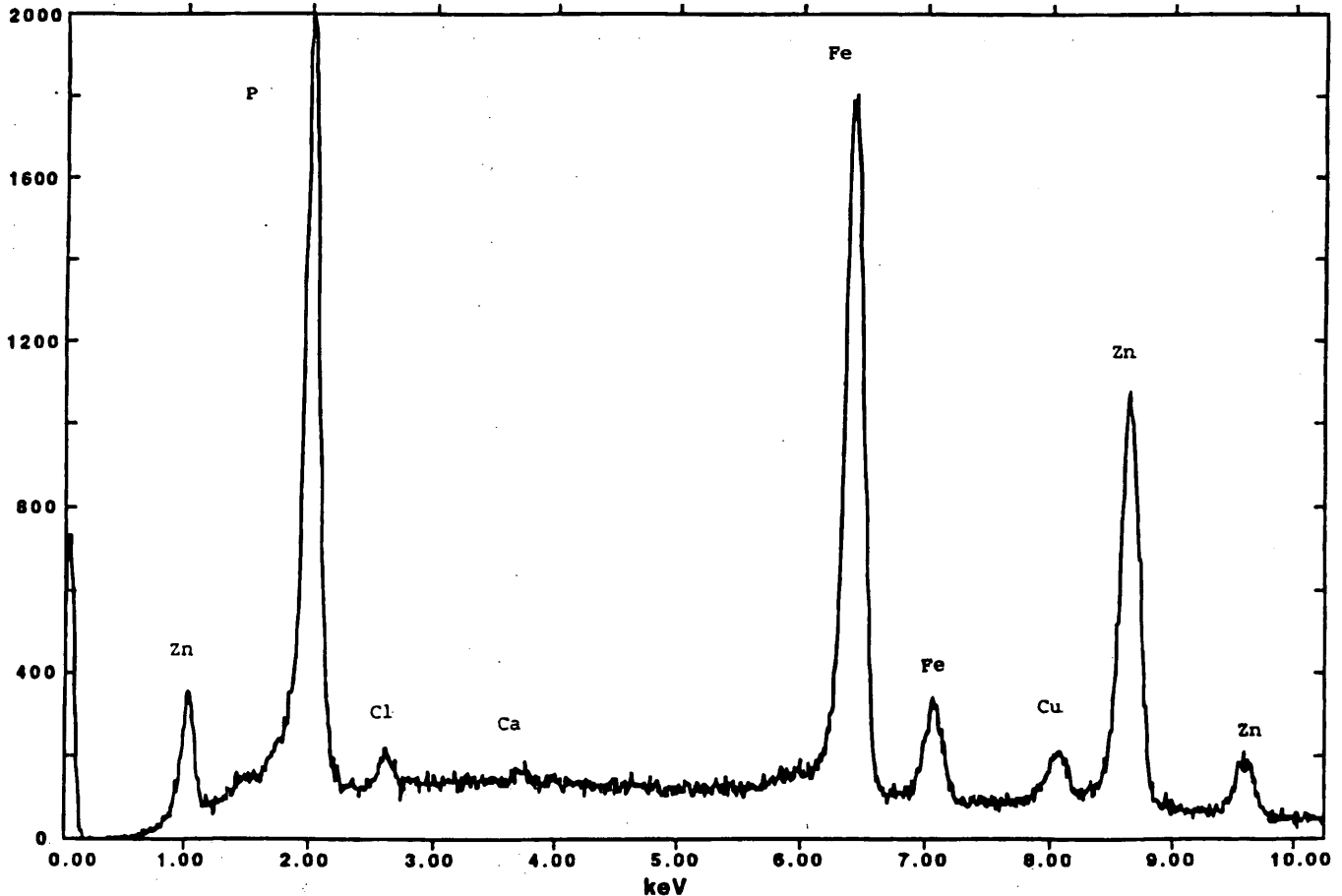


FIGURE 5 EDS elemental analysis of steel surface exposed to a 3 percent solution of salt inhibited with zinc and hexametaphosphate.

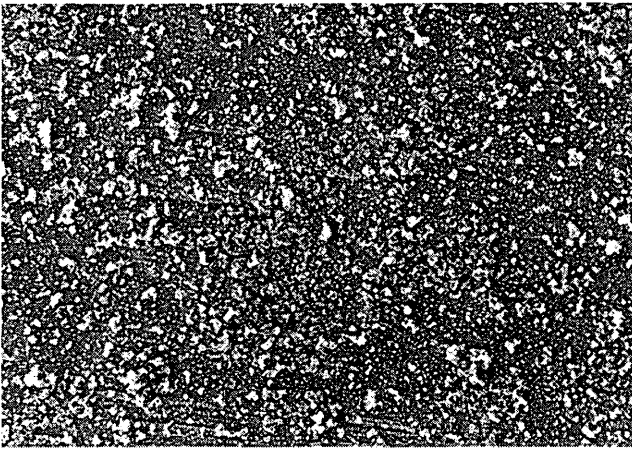


FIGURE 6 Scanning electron micrograph of steel exposed for 1 week to a 3 percent solution of salt inhibited with magnesium and orthophosphate.

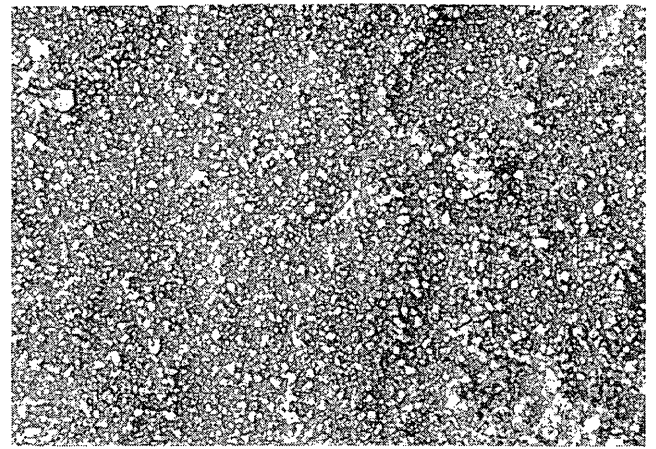


FIGURE 7 Scanning electron micrograph of steel exposed for 4 weeks to a 3 percent solution of salt inhibited with magnesium and orthophosphate.

Surface Analysis

Figure 3 shows the surface of steel exposed to salt solution for 1 week. The electron micrograph shows that as the steel corrodes in salt solution a very porous, flocculent, and mossy-looking layer grows on the salt surface. The EDS elemental

analysis of this surface shows only iron and chloride and is consistent with the formation of a surface layer of iron oxide rust (oxygen does not show up in the elemental analysis because the EDS probe was not sensitive to elements with an atomic number lower than that of sodium) together with some iron chloride. The porous nature of this layer visible in the scanning

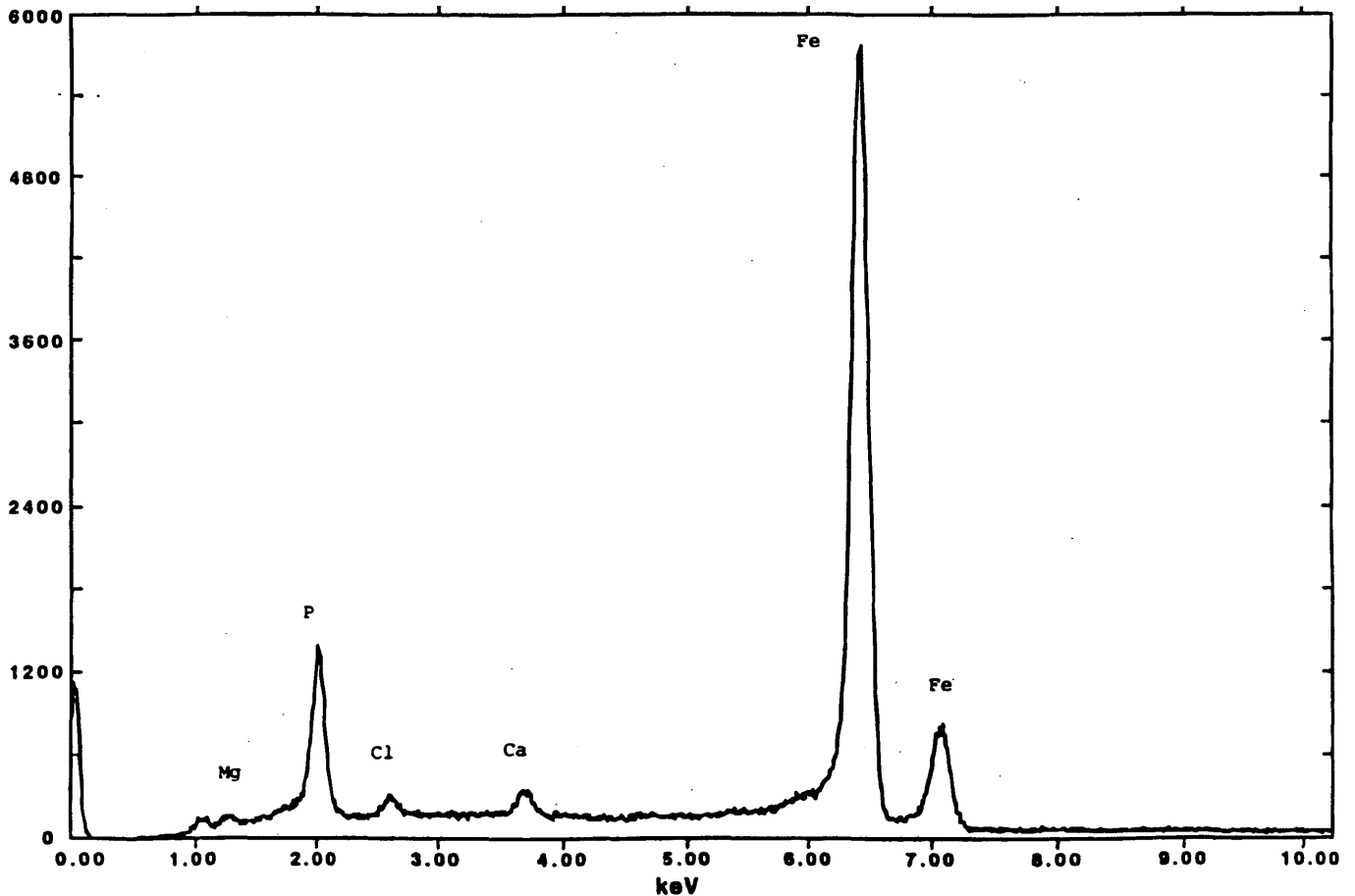


FIGURE 8 EDS elemental analysis of steel surface exposed to a 3 percent solution of salt inhibited with magnesium and orthophosphate.

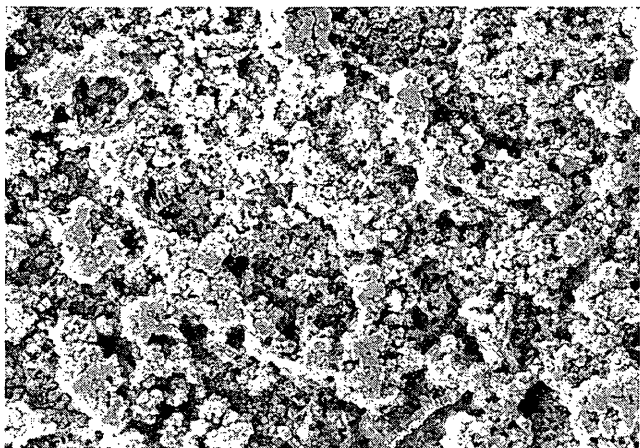


FIGURE 9 Scanning electron micrograph of steel exposed for 1 week to a 3 percent solution of salt inhibited with calcium and phosphonate.

electron micrograph probably permits the easy ingress of oxygen and water to the steel surface, allowing the corrosion reaction to proceed freely.

Figure 4 shows the surface of steel exposed to the zinc-hexametaphosphate-inhibited deicer for 1 week, and it is immediately apparent that a very different kind of surface layer forms. Rather than the voluminous, mossy-looking layer seen in the salt sample, the steel surface in the presence of the inhibitors forms a very compact, flat, uniform layer. The EDS elemental analysis of this surface (Figure 5) shows peaks characteristic of phosphorus and zinc, indicating that the inhibitor molecules become incorporated into the surface layer, presumably causing it to form a dense, compact protective layer. It is reasonable to assume that this flat, compact layer forms a barrier on the steel surface that both prevents the easy ingress of oxygen and water to the underlying

metal and interferes with the diffusion of corrosion products away from the metal surface, resulting in the low corrosion rate indicated in Table 1.

The formation of compact, uniform surface layers into which the inhibitor molecules were incorporated was characteristic of steel surfaces exposed to the orthophosphate- and polyphosphate-inhibited deicers tested in the present study. Another example of this can be seen in Figures 6 and 7, which show the steel surface exposed to the orthophosphate-inhibited deicer for 1 and 4 weeks, respectively. After 1 week so little corrosion has occurred that there is only spotty accumulation of corrosion products on the surface visible on the surface in Figure 6, but Figure 7 shows that after 4 weeks of exposure a compact, uniform sheet has formed over the entire steel surface. The EDS elemental analysis in Figure 8 confirms that the magnesium and phosphorus from the inhibitor are present in the surface layer.

Figure 9 shows the surface of steel exposed to the inhibited deicer containing the low level of phosphonate. It is interesting to note that in this case SEM reveals a surface that looks very similar to that in Figure 3 for plain salt. In both this case and the case of noninhibited salt, the porous, mossy-looking surface layer is associated with high corrosion rates, as shown in Table 1. The EDS elemental analysis of the sample exposed to the phosphonate formula can be seen in Figure 10 and shows only iron, a trace of calcium, but no phosphorus. The lack of inhibitor on the steel surface is consistent with the surface remaining essentially the same as that exposed to plain salt and further suggests that the compact, uniform film formed by the higher levels of orthophosphate and polyphosphate inhibitors is responsible for the low corrosiveness of those deicers.

CONCLUSIONS

Weight loss measurements of the corrosion rate of carbon steel subjected to alternate drying and immersion in 3 per-

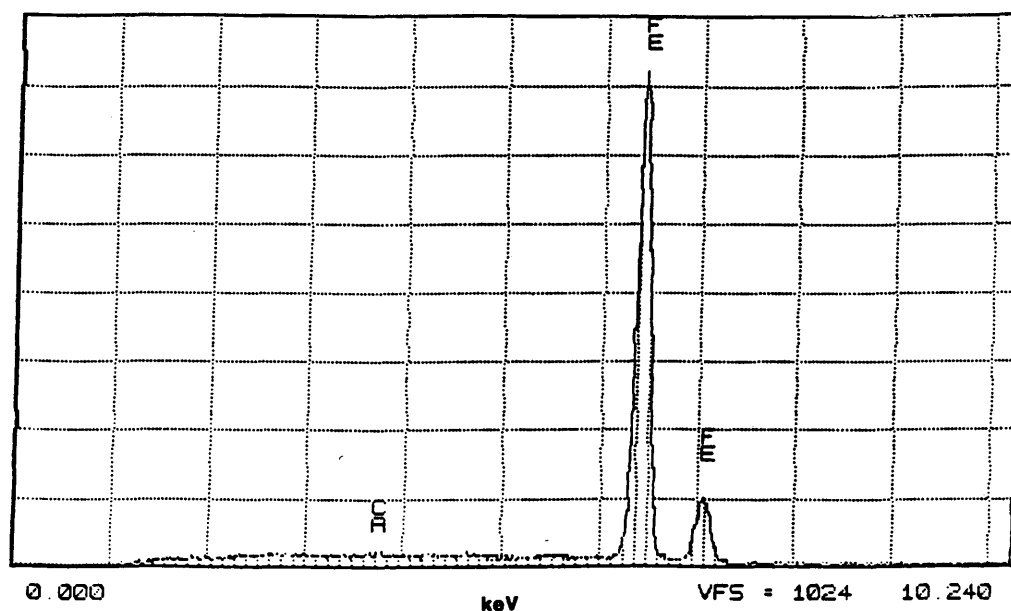


FIGURE 10 EDS elemental analysis of steel surface exposed to a 3 percent solution of salt inhibited with calcium and phosphonate.

cent deicer solutions indicated that the addition of three different phosphates in combination with zinc or magnesium salts effectively reduces the corrosive effects of the chloride ion. The low corrosion rate of salt inhibited with orthophosphate and a magnesium salt was independently verified by linear polarization resistance measurement. The accuracy of the linear polarization resistance measurement in measuring corrosion rate suggests that it can be a useful tool for rapidly determining the corrosiveness of deicers under different concentration, temperature, or length-of-exposure conditions. Phosphate and zinc or magnesium added to deicing salts in amounts of a few percent appear to inhibit the corrosion caused by chloride by becoming incorporated into the surface film that forms on the metal surface. Incorporation of the inhibitor into the surface layer is consistently observed to result in a compact, uniform film rather than the voluminous, mossy-looking oxide deposits seen in the absence of inhibitor or in the presence of a level of inhibitor too low to provide inhibition. This suggests that a primary mechanism for inhibition in these mixtures is through the formation of a protective barrier that insulates the steel from the corrosive environment.

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Publication of this paper sponsored by Committee on Corrosion.