Anodic Protection of White and Green Kraft Liquor Tankage, Part I: Electrochemistry of Kraft Liquors

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Anodic protection is a powerful technique used to mitigate liquor tankage corrosion. The large currents required to protect a typical vessel led to problems in the first generation of commercial systems. This article describes what caused the problems. Part II (March 2002 *MP*) will discuss the solutions developed for successful protection systems.

The kraft pulping process utilizes hot caustic sulfide liquors to remove the lignin that binds the cellulose ($C_6H_{10}O_5$) fibers. The resulting cellulose is used to manufacture many products such as printing paper. The liquors are named white, green, and black, with white being the most concentrated (100 g/L sodium hydroxide [NaOH], 30 gl/L sodium sulfide [Na_2S]).

The first successful use of anodic protection of a kraft liquor tank dates back to December 1984.[1] Since that time, 34 systems have been installed in white and green liquor tanks and clarifiers. During the first several years of commercial application, unexpectedly high corrosion rates were reported at localized areas of several tanks even though the remainder of the surfaces corroded at rates of <5 mpy (127 µm).

This article discusses the most likely causes of high localized corrosion rates and the techniques necessary to prevent such occurrences. Part II, to be published in the March 2002 issue of *MP*, will cover the parameters of anodic protection design and operation.

The electrochemistry of kraft liquors is complex because it involves multiple oxidation states of sulfur compounds (Table 1), numerous possible Fe-S-H_2O reactions, and the existence of active-passive behavior. Crowe[2] published a list of 31 possible Fe-S-H_2O reactions. Crowe and Peterman[3-4] suggest that additional reactions have been reported.

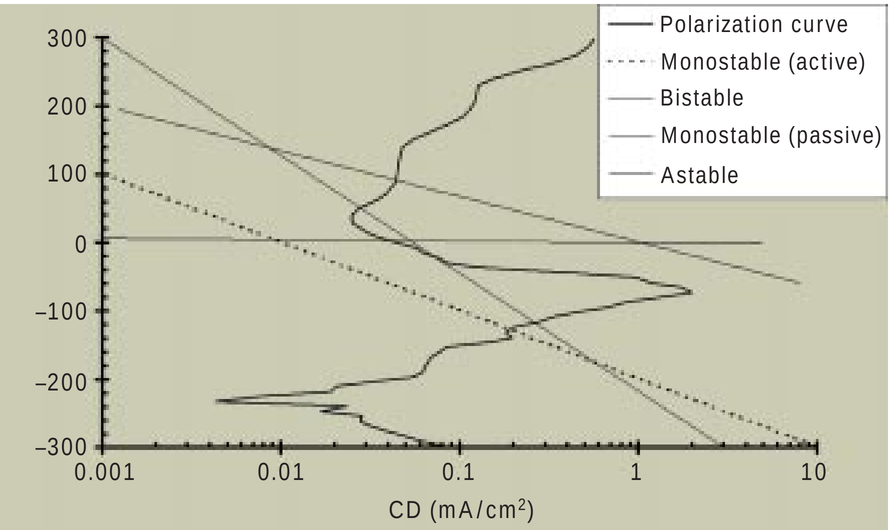
The electrochemistry may be further complicated because some Fe-S compounds are semiconductors.[3] Active-passive behavior requires a negative resistance portion of the anodic curve. Voltage-controlled negative resistance devices are uncommon in disciplines other than electrochemistry. It is interesting to note that a semiconductor device called a tunnel diode

TABLE 1

MAJOR SULFUR SPECIES IN KRAFT LIQUORS

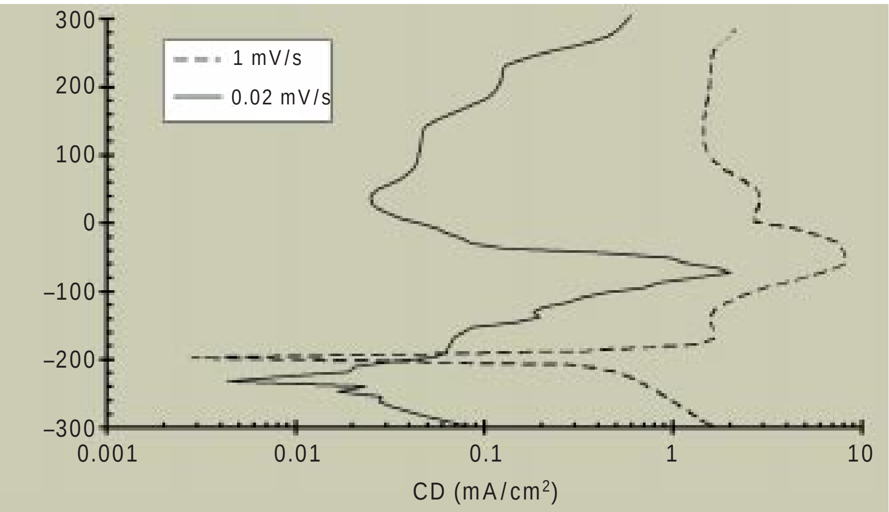
Name	Symbol	Sulfur Valence
Sulfate	SO_4^{-2}	6
Sulfite	SO_3^{-2}	4
Thiosulfate	$S_2O_3^{-2}$	2
Sulfur	S	0
Polysulfide	$S_{x\ (x=1)}^{-1}$	–1
	$S_{x\ (x=1)}^{-2}$	–2
Sulfide	S^{-2}	–2

FIGURE 1



Possible combinations of anodic/cathodic intersections.

FIGURE 2



Typical in situ polarization curve of CS in white liquor at two scan rates.

exhibits behavior nearly identical to that of active-passive interfaces. Additional work will determine if semiconductor films are significant to corrosion studies in kraft liquors.

Tromans[5] deduced a simple model of passivation in caustic sulfide that explains sulfide's role in the process. He concluded that, after the initial nucleation of magnetite (Fe_3O_4), sulfide is incorporated into the lattice. The reaction creates Fe_3O_4-xSx, a nonprotective compound. At the peak of the active-passive transition, Tromans predicted x to be ~0.19. Passivation does not occur until oxidation removes the sulfide from the film. This reaction requires high current densities to proceed, however, once devoid of sulfide, the film will remain stable if the potential is kept more positive than the Flade potential by reducing the reaction of oxidized sulfur species such as thiosulfate ($S_2O_3^{-2}$) and S_2^{-2}.

Figure 1 depicts one anodic and four cathodic idealized polarization curves, including the possible intersection points. The number and location of the intersection points create four types of behavior, namely monostable (active), bistable, astable, and monostable (passive).

Astable behavior occurs infrequently as it requires a single anodic/cathodic intersection on the negative resistance portion of the anodic curve. This unstable operating condition results in continuous oscillations between active and passive potentials. Various alloys in elevated-temperature sulfuric acid (H_2SO_4) exhibit astable behavior.[6]

These four types of mixed-potential models are simplistic and do not accurately reflect the behavior of carbon steel (CS) in liquors because the models assume that the reactions are time-invariant.

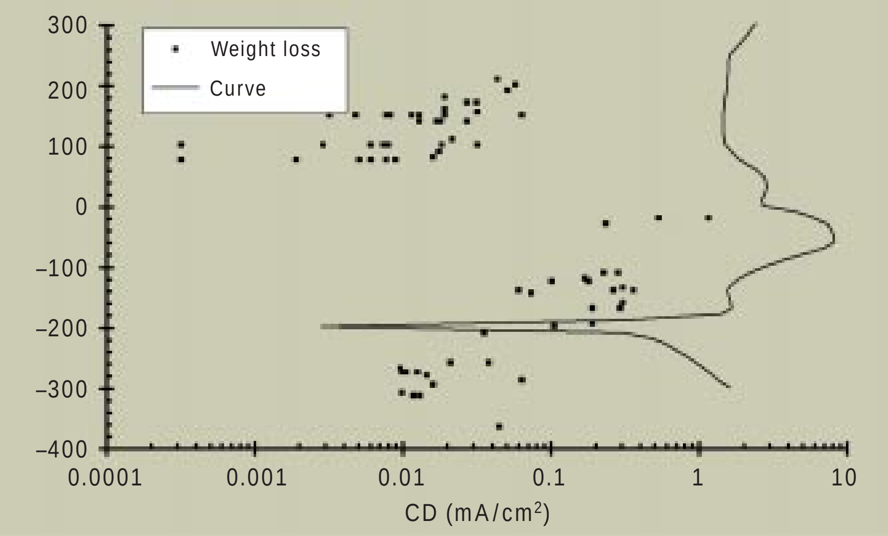
Figure 2 depicts typical curves from an in situ test in a white liquor clarifier at different scan rates. The passive state does not exist until after the active-passive transition is traversed. Therefore, unless CS discharges sufficient anodic current density (CD) via a naturally occurring cathodic reaction or an applied anodic protection current, the CS/liquor interface remains monostable (active) as the passive film and its low CD properties do not exist.

As Tromans predicts and typical curves verify, bistable behavior can occur once the active-passive barrier is traversed. Reversing the direction of the curve establishes a second stable equilibrium potential. Morris[6] used this knowledge to determine protection potentials for stainless steel (SS) and high-nickel alloys in elevated-temperature H_2SO_4.

Anodic Reactions Prior to Traversing the Active-Passive Boundary

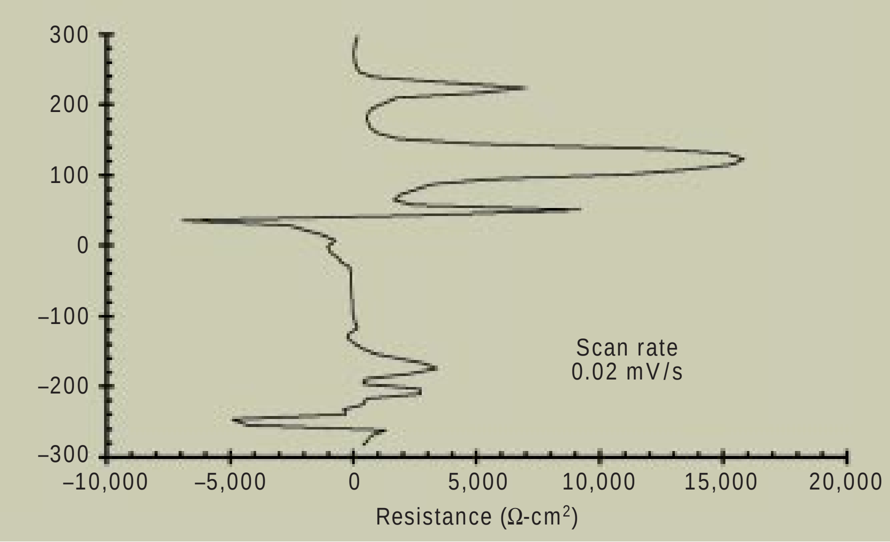
Anodic protection and forced passivity require some finite period of time of operation in the active zone. Thompson[7] performed tests to establish the

FIGURE 3



Coupon weight loss data and polarization curve for CS in white liquor.

FIGURE 4



Film resistance vs potential scan rate 0.02 mV/s.

corrosion rate during these periods. A CS specimen was potential-controlled at the peak of the active zone for 21 h, during which time the current was carefully monitored. The CD averaged 12.5 mA/cm^2. Assuming all of the current caused iron to corrode to the Fe^{+3} oxidation state, the Faradaic loss would have approached 3,800 mpy (96,520 µm/y). The actual measured corrosion rate, however, was only 362 mpy (9,195 µm/y).

Integrating 20 cycles of the anodic curve predicted a penetration of 0.17 mils (4.3 µm) for a single traverse of the active zone. However, the experimental results indicated a value of 0.0135 mils (0.34 µm) per cycle. Hence, iron oxidation accounts for only ~6 to 10% of the discharged current. The remaining current, therefore, must be consumed by oxidation of sulfide in the Fe_3O_4-xSx film or directly from the solution. This produces large quantities of oxidized sulfur species ($S_2O_3^{-2}$ or S_2^{-2}) in the diffusion layer next to the passivated surface. The presence of these reducible ions at the metal/solution interface should significantly increase the cathodic reaction rate.

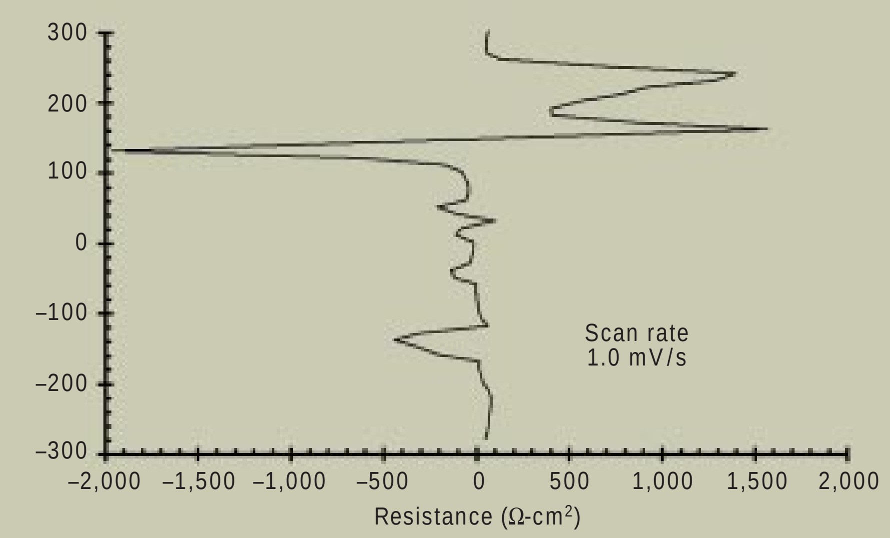
Due to the above situation, the bistable condition that occurs after passivation is not permanently stable. The oxidation of the Fe_3O_4-xSx and enhanced levels of cathodic reactants cause the anodic and cathodic curves to intersect. This lack of stability was verified by passivating a coupon in stagnant white liquor for 3 days, after which time the current was shut off. The coupon's potential remained in the passive zone for 2 days until it reactivated. The cathodic and anodic reactions became unequal so as to create only a monostable (active) operation. Spontaneous bistable conditions do not exist for CS in white liquor. Bistable conditions can occur only after passivation and will not remain if the naturally occurring cathodic reaction rate is less than the anodic reaction rate. Therefore, this bistable condition is better termed "forced bistable."

Cathodic Reactions

In order to control corrosion, an anodic protection system must create and maintain the passive condition under all possible liquor chemistries. Under monostable (active) conditions, the system must provide the required CD to passivate the steel as well as provide the steady-state current necessary to equal the difference in the cathodic and anodic reactions at the optimum potential. If "forced bistable" conditions occur, the system must simply prevent reactivation. The control dynamics for these two cases can differ considerably.

Research has concentrated on the anodic reactions with less emphasis on the cathodic curves. Most work assumes a single cathodic reaction of polysulfide to sulfide in even low-

FIGURE 5



Film resistance vs potential scan rate 1.0 mV/s.

concentration polysulfide liquors. Thiosulfate is a known corrosion accelerator[4,8-17] in white and green liquors, even though direct reduction of this ion is difficult to verify. Peterman[4] concluded that thiosulfate was directly reduced in the corrosion reaction. His data showed that the thiosulfate concentration significantly affected the corrosion potential and corrosion rate.

Figure 3 is a plot of all available potential vs electrochemical equivalent weight loss data.[4,15,17] The conversion assumes that iron corrodes to a Fe^{+3} oxidation state. These data came from many different liquor chemistries and experimental techniques, including potentiostatic control, chemical additions, and open-circuit conditions. It appears that the corrosion rate is much more sensitive to potential than other known variables. Therefore, the corrosion rates in high-content thiosulfate liquor may not be an anomaly because different corrosion products are present, etc. Thiosulfate may somehow affect the cathodic reactions.

Wensley[8] reported that liquors containing high levels of sulfide and thiosulfate passivated CS coupled to SS. Crowe[4] claimed that, although thiosulfate appeared to be an oxidant, it could not passivate CS at any concentration.

Calculations performed by Tromans,[5] from equations provided by Biernat and Robins, may explain the effects of thiosulfate:

$$ES_2O_3^{-2} / S^{-2} = 0.034 - 0.056\ \text{pH} + 0.0093 \log ([S_2O_3^{-2}] / [S^{-2}]^2) \qquad (1)$$

The equilibrium potential ($ES_2O_3^{-2}/S^{-2}$) is –921 mV vs a saturated calomel electrode (SCE) or –21 mV vs a silver sulfide electrode (SSE), assuming an activity of 10^{-6} moles/L for the thiosulfate and 0.423 for the sulfide. This potential shifts ~10 mV in the positive direction for each order of magnitude increase in the thiosulfate activity. Assuming a reasonable Tafel slope for this reaction, an intercept more positive than the critical potential for passivation of CS is not possible. Hence, increases in the concentration of thiosulfate can increase the corrosion rate but cannot passivate CS. At passive potentials, thiosulfate may be oxidized and hence play no major part in the reduction reaction.

Estimating[18] the equilibrium potential of S_2^{-2}/S^{-2} puts it at ~100 to 150 mV vs SSE. Polysulfide reduction, therefore, can provide sufficient current to passivate CS if enough polysulfide is present in the solution.

Separate Active and Passive Areas in Anodically Protected Tanks

As a result of the high conductivity of white liquor, several "myths" surround the use of anodic protection. One myth is that active areas will be galvanically polarized passive if the majority of the surface is passive. The forced bistable scenario, can facilitate the coexistence of stable passive and active intercepts; the intercepts.

The simultaneous existence of separate active and passive areas generates low and marginally higher-than-open-circuit corrosion rates, respectively. This type of operation is far from optimum. It is not, however, the worst possible scenario. Figures 4 and 5 indicate the effective film resistance for a sample of 1 cm² as a function of potential. The resistance is calculated from the slope of the polarization curves. The resistance is low in the active zone and negative from the current peak to the Flade potential. It increases to large values in the passive zone. The film resistance in the passive zone is time-dependent.

The potential measured by a reference electrode is the weighted average of all potentials of a structure. The weighting factor is the resistance (solution and film) from the location of the various surfaces to the reference. The situation is complicated by an IR voltage drop created by protection current flowing through the electrolyte. As a result of the large differences in surface resistance between active and passive areas, an active area is detectable by a reference at any location in the structure.

If an active area develops, the potential controlled current source (potentiostat) provides more current in response to the lower reference potential. Unfortunately, the IR at the reference location increases the measured potential. It masks the evidence of the active area, and the source produces insufficient current to passivate the zone. Without sufficient CD to be passivated, small active areas increase in size and produce a larger current output from the power supply on the active site. An equilibrium eventually forms between the size of the active area and the current output. This causes a location (usually an ellipse) to operate at the peak anodic CD of CS in liquor, hence causing very high corrosion rates.

System operation in this mode is easily corrected once the possibility of coexistence of active and passive zones is verified. The potential of an active area is so dominant because of the low resistance to the reference that it can be easily detected. Passivating an active area is simple if detected and repassivated when the area is small.

Potential Measurements in Anodically Protected Tanks

Extensive potential data have been collected from ~40 white and green liquor tanks. Unprotected potentials of all tanks except two were –125 ± 10 mV vs SSE regardless of liquor type (white and green). One of the anomalous tanks indicated potentials of –110 and –80 mV at opposite points on the shell circumference. The potential of the other tank, which was fabricated using large amounts of SS, was in the passive zone (75 mV vs SSE).

The most frequent potential range of –125 mV corresponds within the accuracy of a SSE to the minor peak at –770 mV (vs standard hydrogen electrode [SHE]) reported by Tromans.[5] He concluded that this was the potential at which S^{-2} ions incorporate into the lattice.

Potentials in the immersed liquid/air interface are normally 5 to 10 mV more positive than at the bottom of the tanks. The distribution of potentials is linear with liquor depth. No abrupt change occurs at the interface between the wet/dry and constant immersed zones.

If a passivation attempt is aborted, the potentials move 10 to 20 mV more negative than the "static" values. This change has remained for as long as 2 months.

If the anodic protection system is turned off, the potentials decay in the negative direction in two discrete steps. The initial step is rapid, decaying to a value of ~ –75 mV vs SSE. This potential remains for days and, in one case, lasted 3 weeks. The next step occurs over hours and is linear with time to a potential of ~ –125 mV vs SSE. These decay times correspond to the effective film resistance vs potential. Unlike initial passivation, the time required to reestablish the set potential is relatively short (30 min), suggesting that the passive film was still intact. Perhaps reincorporating S^{-2} back into the film is irreversible or, more likely, very time-dependent.

When anodically protected tanks are cleaned of lime mud and iron sulfide (FeS) deposits, the underlying surface exhibits a bronze-colored hue. This thin film is thought to be either pyrite (FeS_2) or $NaFeS_2 \cdot 2H_2O$.

Summary

Most of the problems encountered with anodic protection of liquor vessels so far have resulted from an incomplete understanding of the electrochemistry of CS in liquors and the assumption that the current distribution was secondary and not primary. This caused the creation of active and passive coexisting areas. Earlier control philosophy did not account for the possibility of active and passive coexisting sites. Present systems recognize and avoid these problems.

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Anodic Protection of White and Green Kraft Liquor Tankage: Part II—Anodic Protection Design and System Operation

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Anodic protection is a powerful technique used to mitigate liquor tankage corrosion. The large currents required for protection of a typical vessel led to problems in the first generation of commercial systems. Part I of this article (February 2002 *MP*) describes the cause of the problem. This article discusses the solutions developed for successful protection systems.

Anodic protection and control system designs now incorporate all of the features required to passivate a tank, maintain passivation, detect active areas, and repassivate if required. Figure 1 shows the general layout of the original type of system.

Current Distribution in Liquor

White liquor exhibits a resistivity of ~1 Ω-cm,[1] which is 30 times more conductive than seawater. This high conductivity, coupled with the previously discussed large resistance of the passive film (Part I, February 2002 *MP*), can make one think that the current distribution in liquors is uniform regardless of the location of the cathodes. This incorrect assumption can lead to incomplete passivation of liquor tanks.

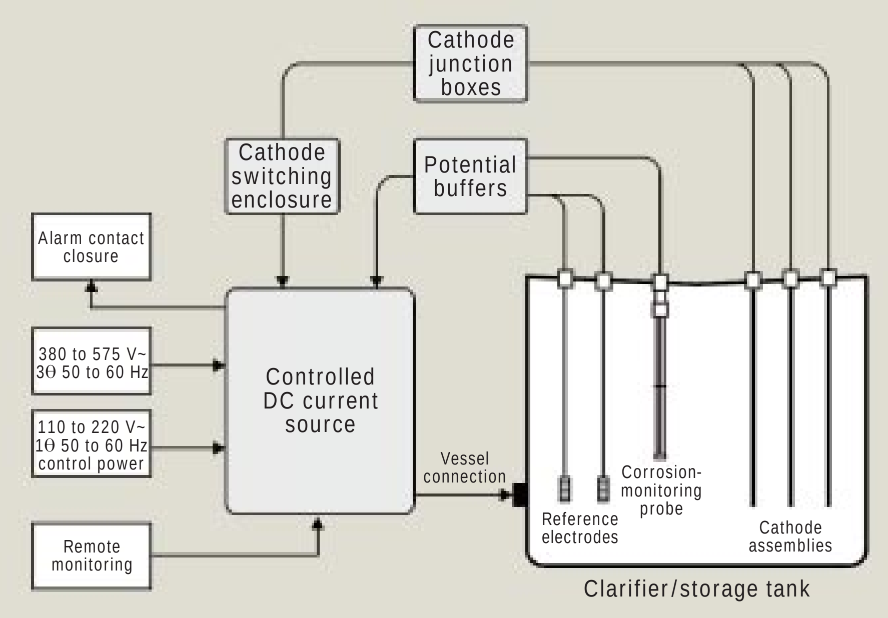
Primary current distribution is solely a function of the geometries of the anode and cathode. All surfaces are considered to have the inability to polarize, so solution conductivity and electrode kinetics are eliminated from the calculations. Figure 2 depicts the primary current distribution calculated for a line-plane geometry. As the line electrode approaches the plane electrode, the current uniformity on the plane decreases in a nonlinear manner.

Secondary and tertiary current distribution assumes electrode polarization. If the electrochemistry of carbon steel (CS) in kraft liquors resulted in only a single current peak at the active-passive transition, followed by a low current zone, then polarization of the tank surface would yield very uniform current distribution. In white liquor, the sulfide breakaway current prevents significant secondary distribution (Figure 3). Sulfide oxidation consumes the excess current density (CD). Hence this current cannot be distributed to other locations. Anodic protection of liquor tanks, therefore, must be designed on the basis of primary—and not secondary—current distribution.

Cathode Design

The location of cathodes affects the current distribution to the tank walls. Design is based on primary current distribution, with the ratio of the minimum

FIGURE 1



Anodic protection electrical block diagram (single clarifier/storage tank).

to maximum CD around the circumference of the tank >0.9.

The design of the system must allow for a fluctuating liquor level. This requires higher initial CD and more frequent repassivation cycles to form a tenacious passive layer. When immersed, the wet/dry zone of a tank exhibits a more positive potential than the remainder of the tank, which may account for the higher corrosion rates in this area. It has been observed, however, that the wet/dry zone remains uncovered by surface buildup or deposits. The constantly immersed zone builds a thick surface deposit on these protected surfaces. All of the electrochemistry discussed in Part I also applies to decanted liquor. Surface buildup probably diffusion-limits all of the reaction rates.

CONTROL SYSTEM

Conventional control schemes for electrochemical protection systems rely on a simple proportional, integral (PI) algorithm. This technique is not optimal when active and passive areas exist simultaneously. Using this type of control has caused accelerated corrosion of active areas.

When a surface is entirely passive, the measured potential is uniform to within a few millivolts around the circumference of the tank. An active area of any size significantly distorts this uniformity. The measurement is so precise that active areas can be located by triangulation.

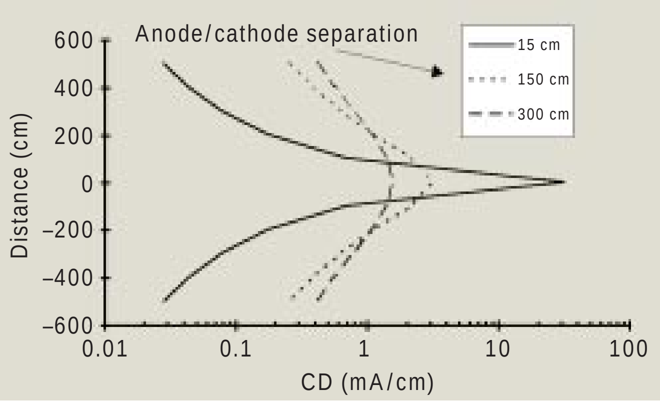
Once this distortion is detected, the control system initiates an automatic repassivation using high currents for a programmable period of time. Once repassivation is complete, the system is retested to ensure uniform passivity.

Implementing this scheme has revealed when and where active areas are formed on the vessel. Once the cathode design has been modified to provide uniform distribution of current, all activations have occurred only at the wet/dry zone soon after initial passivation. The initial stability of the passive film in this zone is less than at other locations. Several repassivations are common during the first month of operation. Activations seldom occur after this stabilization period.

Corrosion Rate Measurements of Protected Tanks

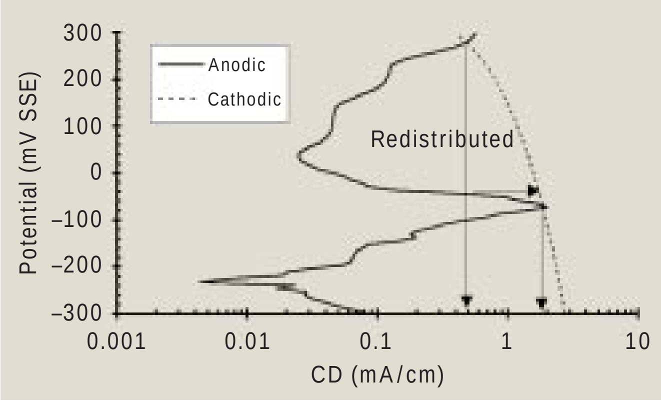
After discovering the possible problems affecting anodic protection of tanks and clarifiers, it was necessary to

FIGURE 2



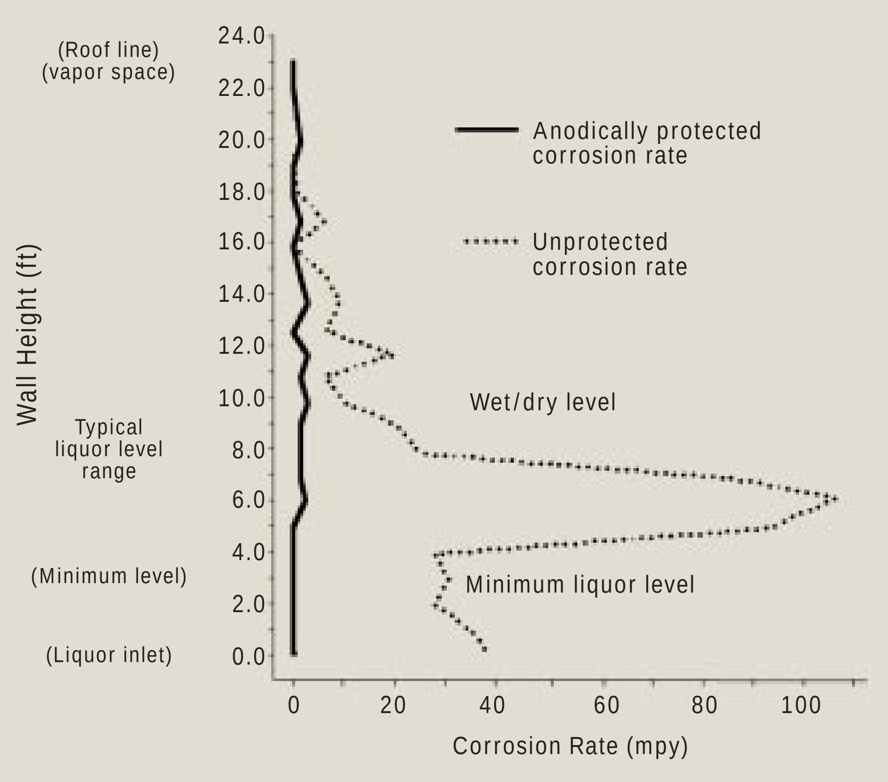
Current distribution (primary) line-plane model.

FIGURE 3



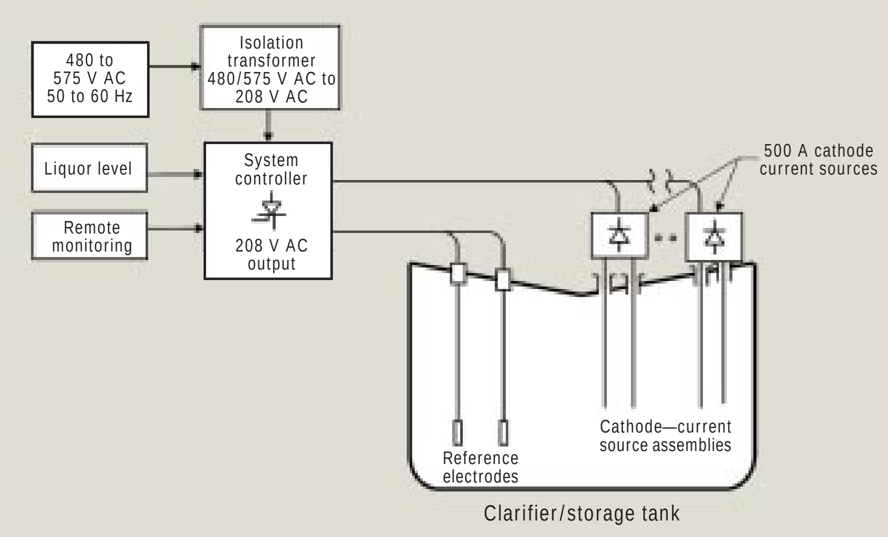
Effect of sulfide oxidation on current distribution.

FIGURE 4



White liquor clarifier corrosion rates.

FIGURE 5



Anodic protection electrical block diagram using distributed current source.

verify that the implemented solutions lead to significant reductions in the corrosion rate in both the wet/dry and immersed zones of tankage. Extending multiwire coupons from the top to the bottom of the tank is one way to do this. A direct connection to the tank protects one of the wire coupons while the second is isolated to simulate unprotected corrosion rates. A third stainless steel wire supports the other wires.

These coupons were originally 3.17 mm in outside diameter (OD), which proved to be a sufficient cross-sectional area to duplicate corrosion rates in unprotected tanks. The coupons appear to corrode at a slightly higher rate than the actual tankage in the immersed zones—probably because of lime mud effects. The wet/dry zone, however, gives results that correlate well with actual tank thickness loss.

As a result of the high CD required to initially passivate a vessel and attached coupons, the 3.17-mm OD coupons proved to have too large a lineal resistance to ensure passivation. Coupon diameters have since been increased to ensure they accurately model the tank. Figure 4 displays typical corrosion rates for protected and unprotected coupons.

In addition to coupon testing, several tank shells were thickness-tested by carefully taken ultrasonic readings. One of the tanks that had previously experienced accelerated active corrosion was tested internally using a 2-by-2-ft (0.6-by-0.6-m) grid. The results indicated an average corrosion rate of <6 mpy (152.4 µm/y).

In a clarifier being monitored by both internal and external ultrasonic thickness measurements, the corrosion rate appeared to be well under 10 mpy (254 µm/y).

Cost Reductions of Installed Systems

Most costs associated with the early anodic protection systems for liquor vessels stem from the power supply and installing the heavy-capacity cable needed to transmit high-current, low-voltage direct current (DC) power from the controlled current source to the tank. With this system design, the installed ampacity of the rectifier was ~50% of the current required to passivate a full-operating tank. As such, passivation required that the tank level be reduced to <50% of the full level.

The immersed surfaces were protected, after which the liquor level was slowly raised. Assuming a sufficiently slow fill rate, the newly immersed tank wall area would be passivated.

If the liquor was added too quickly, an active zone would form and the level would once again have to be lowered to a point where total passivation was reestablished.

This arrangement was a problem because the plant operators required the flexibility of using the tank storage capacity as a buffer if a process upset occurred somewhere in the pulp mill. This resulted in rushing the passivation, which contributed to the coexistence of active and passive areas in the tank, as previously discussed.

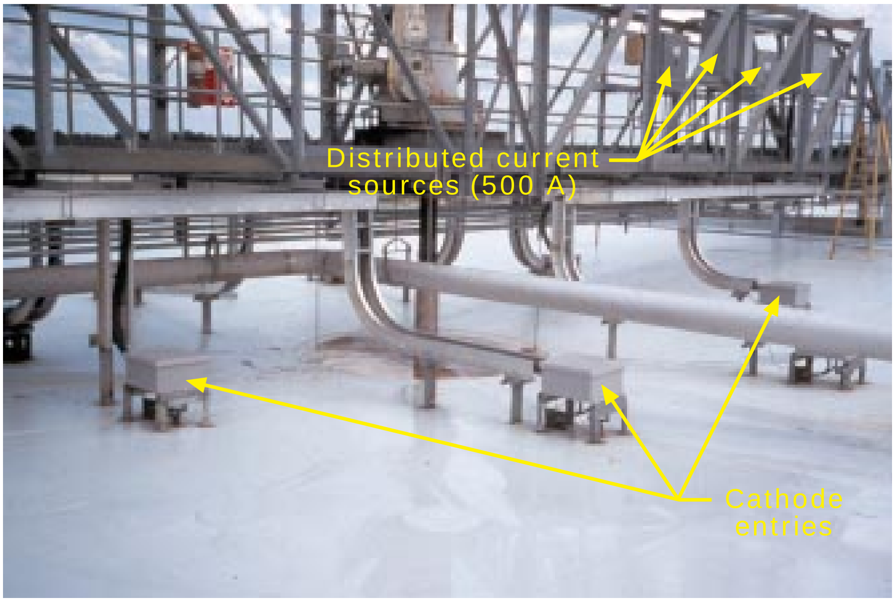
Since 1994, system design has significantly changed to overcome the shortcomings of the early systems with no increase in installation cost. Figure 5 shows the new arrangement using distributed current sources for each cathode. These sources are installed on top of the tank; hence, the heavy DC cables are kept short.

The system is controlled by a 208-V alternating current (AC) 3-phase controller located in a motor control center. As the power control is achieved at 208-V AC 3-phase (as opposed to 12 V DC), the interconnecting wiring can be downsized to significantly reduce the installation cost. The systems are now supplied with 100% of the current required to passivate a nearly full vessel, minimizing the need to lower the tank level during passivation. Figures 6 and 7 are photographs of the system arrangement.

FIGURE 6

Distributed current source arrangement.

FIGURE 7



Distributed current source arrangement and cathode entries.

Conclusions

- Under normal operating chemistries in white and green kraft liquors, CS exhibits a monostable (active) behavior. Corrosion rates depend on the presence of reducing agents (or cathodic reactions).
- Bistable behavior occurs only after the passivation process has reached some degree of completion.
- Once created, the passive state is not spontaneously permanently stable.
- During traverse of the active zone, the corrosion rate is ~10% of the calculated Faradaic equivalent; hence, 90% of the current is consumed in sulfide oxidation.
- The role of thiosulfate ($M_2S_2O_3$) in the corrosion process requires additional research.
- Active and passive areas can coexist.
- Cathode locations in kraft liquor anodic protection systems must be based on primary current distribution.

- Operating tanks exhibit corrosion potential values in the range of –125 mV vs silver sulfide electrode (SSE), regardless of liquor type.
- Anodic protection reduces the corrosion rates significantly once the system is designed to account for kraft liquor electrochemistry.
- Anodic protection systems are now designed to passivate vessels with minimal changes to liquor level.

Acknowledgments

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