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METALLURGICAL ANALYSIS OF LEAKING TRAY PACKS

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ABSTRACT

A metallurgical analysis was performed to determine the cause of leaking tray packs and the origin and development of "gray spots" which represent potential leak sites. Tray packs are fabricated of tin-free steel (TFS) and are protected with multilayered coatings on both interior and exterior surfaces. Accelerated corrosion tests were carried out in order to monitor the progress of corrosion in a simulated long-term exposure environment. These tests exposed defects in the interior coatings (pores, cracks, blisters) which led to pitting corrosion of the TFS within a few days of exposure to the chloride environment. The presence of "gray spots" on the exterior surface of the tray packs was related to an existing defect in the directly opposite surface of the interior coatings. Microscopic examination, in conjunction with AC impedance measurements, showed that water and aggressive ions contained in the food penetrate into and through interior coatings, through localized defects such as pores and cracks, causing pitting of the TFS which intensifies as a result of the presence of an occluded electrochemical cell at the coating/metal interface. Once the pit has penetrated through the entire thickness of the TFS, a gray spot appears under the exterior clear epoxy coating. AC impedance measurements also were made to compare the corrosion resistance of the current multilayered coating system with a newly developed single phase coating (HGW). The greater values of polarization resistance derived from these measurements indicated that the current multiphase coating system has a higher degree of corrosion resistance than the new HGW coating.

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INTRODUCTION

Reports of leaking tray packs were received during the period of 1984 through 1987. On-site investigations confirmed these reports and noted the presence of "gray spots" on the exterior surface of some of the tray packs. If probed gently with a needle or similar object, some of these "gray spots" develop into leak sites. In order to complete execution of contracts requiring delivery of about one million tray packs, the "gray spots" problem had to be resolved and a high level of confidence developed in the ability of the contractors to deliver tray packs with acceptable low defect rates and to meet the self-life requirement. Accordingly, the Natick Research Development and Engineering Center (NRDEC) developed a comprehensive action plan, which included Materials Technology Laboratory (MTL) participation, in determining the origin and development of gray spots and assessing new coatings being considered as replacements for the current coating system. The stamped tray pack is fabricated of tin-free steel (TFS). The interior of the tray pack is coated with a multilayered system comprised of Cr, Cr_2O_3, epoxy and, finally, vinyl. The exterior surface is coated with Cr, Cr_2O_3 and a clear epoxy. This report presents results of the "gray spots" investigation and compares the corrosion resistance of a new proposed proprietary single layer (HGW) coating with that of the currently used multilayer coating system.

EXPERIMENTAL PROCEDURES

The current coating system for tray packs is shown schematically in Figure 1. Accelerated salt water/salt fog corrosion tests of a virgin and an emptied food-containing tray pack were carried out in order to monitor the progress of corrosion in a simulated long-term exposure environment. The observed localized corrosion was to be related to existing coating defects and the presence of gray spots (see Figures 2a and 2b). In addition, numerous defective tray packs were examined metallographically by optical and scanning electron microscopy (SEM) for determination of the operating mechanism of coating degradation. Further, AC impedance (electrochemical impedance spectroscopy) was employed to compare the corrosion resistance of the current coating system versus a new single phase coating in a chloride solution.

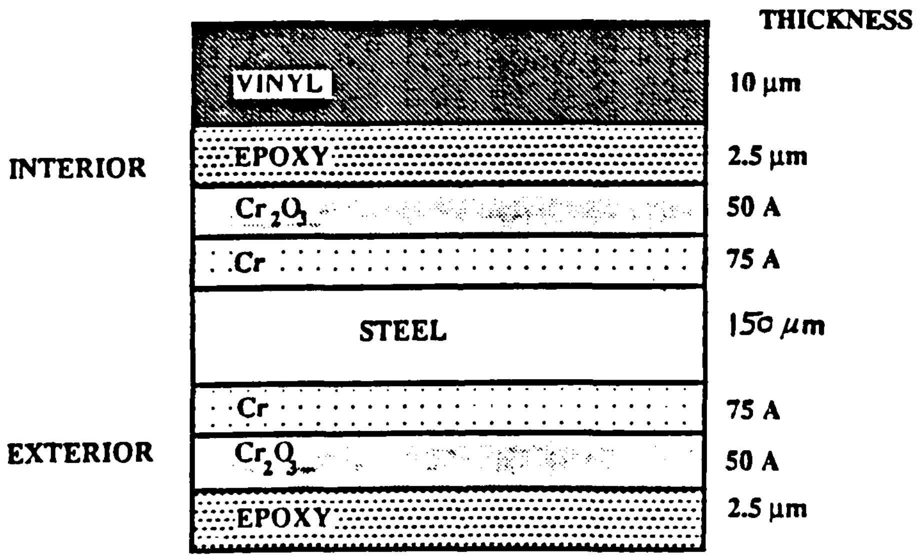
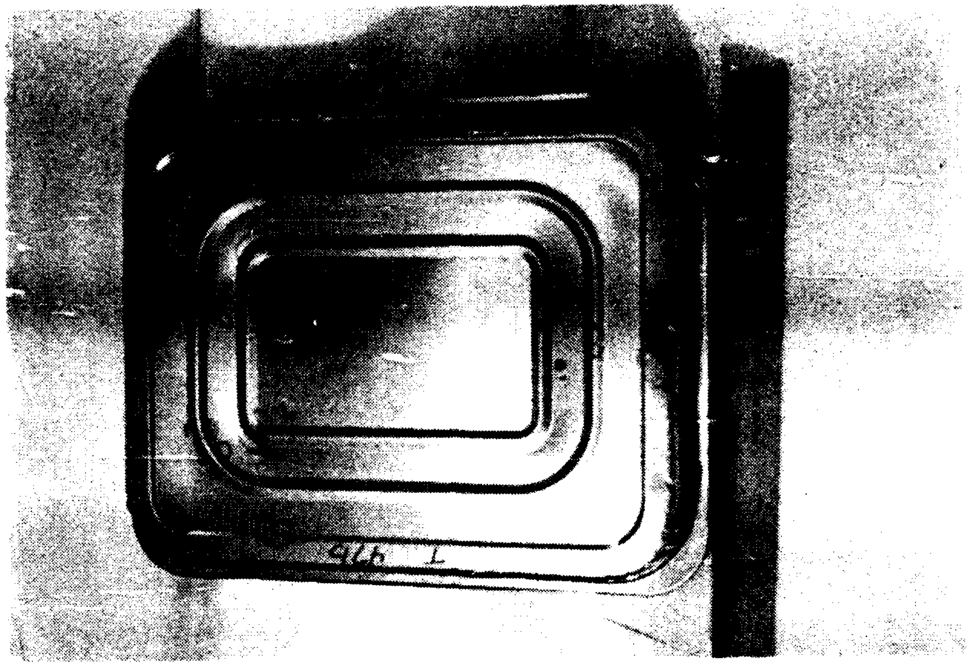
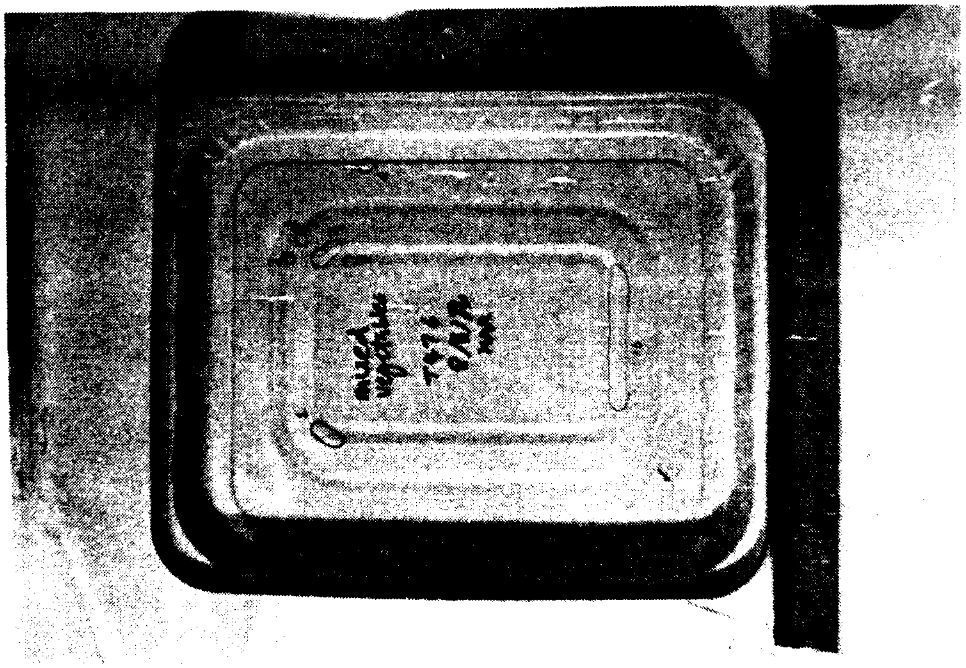


Figure 1. Schematic cross-sectional representation of coating layers and thicknesses on the tin-free steel used for food tray fabrication.



(a) Exterior



(b) Interior

Figure 2. Circled "gray spots" on the exterior and interior of used tray (T476).

RESULTS AND ANALYSIS

Salt Water Immersion

One virgin tray was immersed in five percent sodium chloride solution at room temperature, and the development of rust was monitored for six days. Five defects on the interior (white vinyl) coating were initially observed before the tray was placed in the salt water. These defects were in the form of scratches due to prior handling since the interior white vinyl layer is very soft. Typical red oxide normally associated with rusting developed along one of the defects after only one day of immersion. After four days, rust was observed on two more defects which were located on the highly strained areas of the tray pack. Rust was also found on the edges which are not normally coated. There was no rust on the exterior surface which was also observed to be covered with a film of unknown protective grease.

Salt Fog

One tray (MI96) dated February 25, 1987 was opened and the contents consisting of green beans, were removed. This tray was cleaned with soapy water and subjected to salt fog testing in five percent NaCl according to ASTM B 117. Several gray spots were observed on the exterior surface of the tray before the test. The exterior was exposed first, and after one day of exposure, large areas of rust developed. These rust areas were distributed evenly over the entire surface, but did not necessarily correspond to gray spots. Then the tray was inverted and the interior was exposed to salt fog. After one day, several rust spots were observed. These rust spots increased in number and became heavier after five days of exposure. Most rust spots appeared at the highly stressed areas. Another coated tray (consisting of beef stew, March 5, 1983, W215) was subjected to similar testing after the contents were removed and similar results were obtained.

These accelerated tests, immersion in NaCl or salt fog, showed that chloride and oxygen ions can permeate through the interior protective coatings and react with the steel to form rust. The observed corrosion appears to occur primarily at highly stressed areas.

Visual Examination and Optical Microscopy

Sixteen trays previously containing various food stuffs were visually examined. Gray spots and pin holes were found on 11 of them regardless of the contents (see Table 1). One gray spot was probed with a sharp pencil and examined under the optical microscope (see Figure 3). There was no evidence of corrosion products in the probed area. Four tray packs with gray spots (two contained cut green beans, one contained mixed vegetables, and one contained creamed ground beef) were selected for closer examination under the optical microscope. These trays were opened and the contents removed. The containers were then cleaned with soapy water, sectioned, and examined. Figure 4 is an example where all the gray spots on the exterior of the tray had corresponding coating defects inside the tray, but not all the interior coatings defects had corresponding gray spots on the exterior surface (see Figure 5). The average size of the gray spots is 0.5mm in diameter, and the corresponding coating defects are always larger. The defects on the interior coating that correlate with gray spots exist as either blisters (see Figure 6) or pits. The pits appear to be associated with the tray pack containing meat, while blisters were found only on the vegetable-containing tray packs. The blistering between primer and topcoat, or through both coatings, may be due to attack by water and/or salt solution via electrochemical action at cathodic areas of the TFS where an alkaline reaction occurs ($O_2 + H_2O + 4e^- \rightarrow 4OH^-$).

Table 1. VISUAL EXAMINATION RESULTS OF FOOD TRAY SAMPLES

Sample No.	Contents	Date Pack	Date Open	Observations
T126	Green Beans		Jul-87	Many Rusts on Outside
M193	Green Beans	2/25/87	Jul-87	Some Gray Spots Outside
V001	Virgin			No Defects
T113	Beef Slices with BBQ	2/7/84	Jul-87	Rust on Outside, Color Changes Inside
W215	Beef Stew	3/5/83	Jul-87	Gray Spots and Pin Holes
N001	Unknown			Gray Spots and Blisters
M196	Green Beans	2/25/87	Jul-87	Gray Spots and Coating Damages
V002	Virgin			No Defects
T164	Beef Stew	11/3/83	Jul-87	Punched Through Holes
1071-18A, T155	Apple Dessert	7/4/84	Jul-87	No Defects
1071-16C, T134	Cut Green Bean	3/7/84	Jul-87	Many Small Pin Holes and Gray Spots
1017-16B, T132	Cut Green Bean	3/7/84	Jul-87	Some Pin Holes Inside and Gray Spots Outside
T476	Mixed Vegetables	3/8/84	Jul-87	Many Pin Holes and Gray Spots
1017-19B, T144	Creamed Ground Beef	10/26/84	Jul-87	3 Pin Holes and Gray Spots
D	Ham Slices in Brine	5/31/84	Nov-87	Gray Spots with Corr. Defects
E	Beef Stew	4/22/83	Nov-87	5 Gray Spots with Corr. Defects
FM2283	Frankfurters in Brine	8/16/83	Nov-87	No Gray Spots, 4 Corr. Defects Inside with Rust
J	Lasagna with Meat Sauce	7/26/83	Nov-87	No Gray Spots, No Defects, Some Rust

Scanning Electron Microscopy

In order to elucidate the gray spot morphology, SEM examination was carried out on an interior coating blister area (see Figure 7a) which corresponded to the gray spot on the exterior surface of the tray. By repeated application of acetone and methyl ethyl ketone to one of the blisters, it was possible to remove the vinyl and epoxy coatings and reveal the pit which had formed in the steel (see Figure 7b). A final ultrasonic treatment also removed the exterior epoxy coating exposing a through-hole, as shown in Figure 8. The pit was hemispherical in shape and a mud crack pattern, typical for an iron oxide, was observed on the wall of the pit (see Figure 9).

It should be noted that since the white vinyl coating is relatively soft, it is susceptible to mechanical damage and is then permeable to water. The epoxy coating beneath the vinyl is hard and brittle and contains internal stresses generated during the curing cycle. Also, the coatings are applied to the tin-free steel sheet prior to stamping into the desired tray pack form. Because of the nature of the epoxy, it is likely that the stamping operation will cause cracks to form in the epoxy coating particularly in areas of high stress. These cracks could be concealed by the soft ductile vinyl overlay.

In order to confirm the above, the epoxy layer was examined by SEM after the vinyl overlay was removed chemically with acetone and methyl ethyl ketone. Figure 10 shows that many small cracks 10 to 15 μm long are present in the epoxy coating. These cracks are located in highly stressed areas (corners, edges, and sidewalls) and are aligned in a preferred orientation corresponding to a circumferential direction.

(a) Exterior

(b) Interior

Figure 3. Optical photomicrographs of a pencil probed 'gray spots' on the exterior and interior of tray (T312).

(a) Exterior

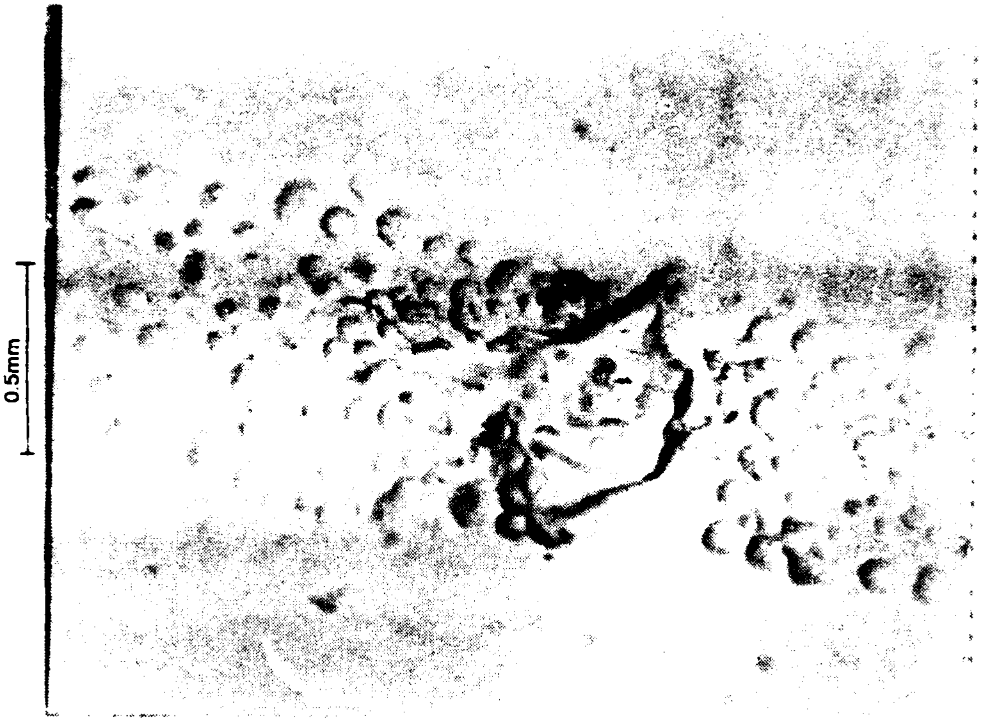
(b) Interior

Figure 4. Corresponding "gray spots" on the exterior and coating defects on the interior of tray (T476).

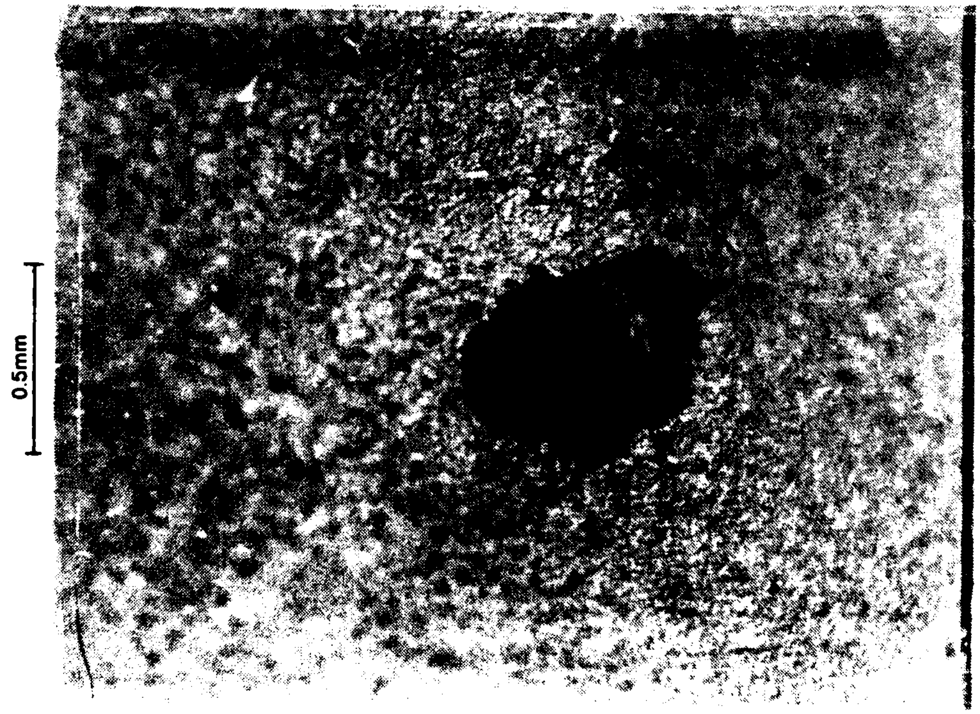
(a) Exterior

(b) Interior

Figure 5. A half-thru pit normal appearance w/o defect on the exterior and a pit observed at the opposing position on the interior of the tray (T144).

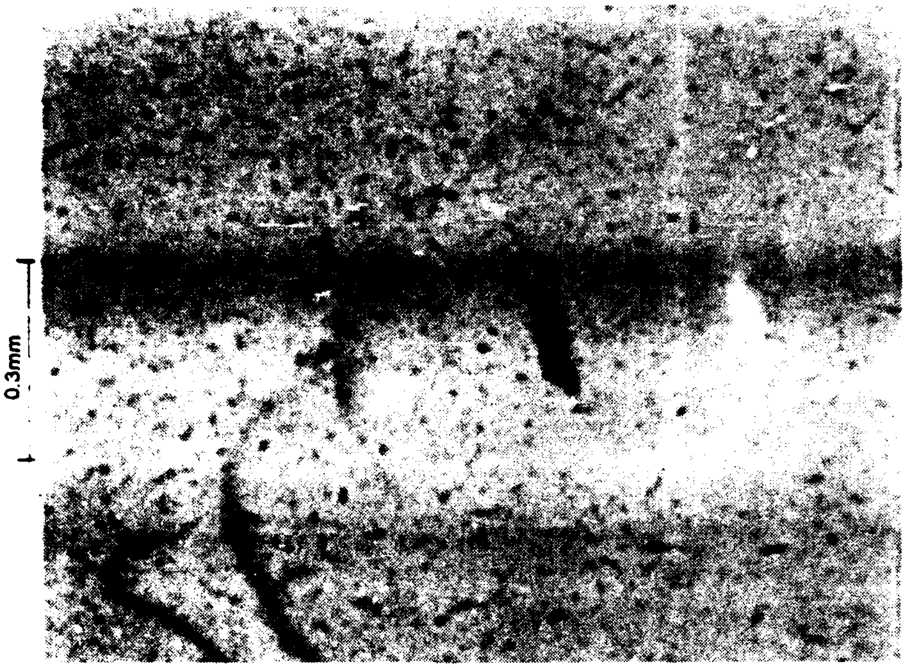


(a) Blisters



(b) Pits

Figure 6. Blisters and pits on the interior surface corresponding to the 'gray spots' on the exterior side of the tray.



(a) Before

(b) After

Figure 7. A pit covered by a broken sheath on the interior of a tray before and after acetone wash (T134).

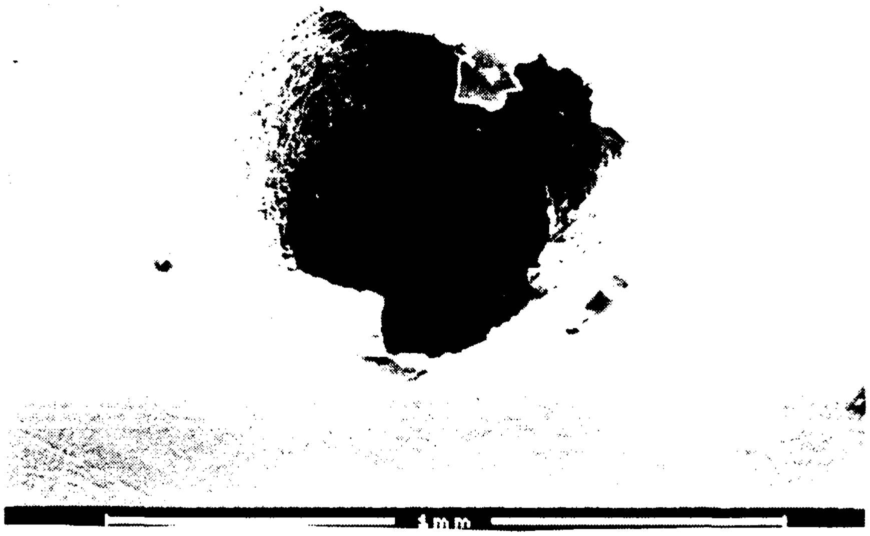


Figure 8. A thru-hole pit on the interior of a tray after ultrasonic cleaning (T134).

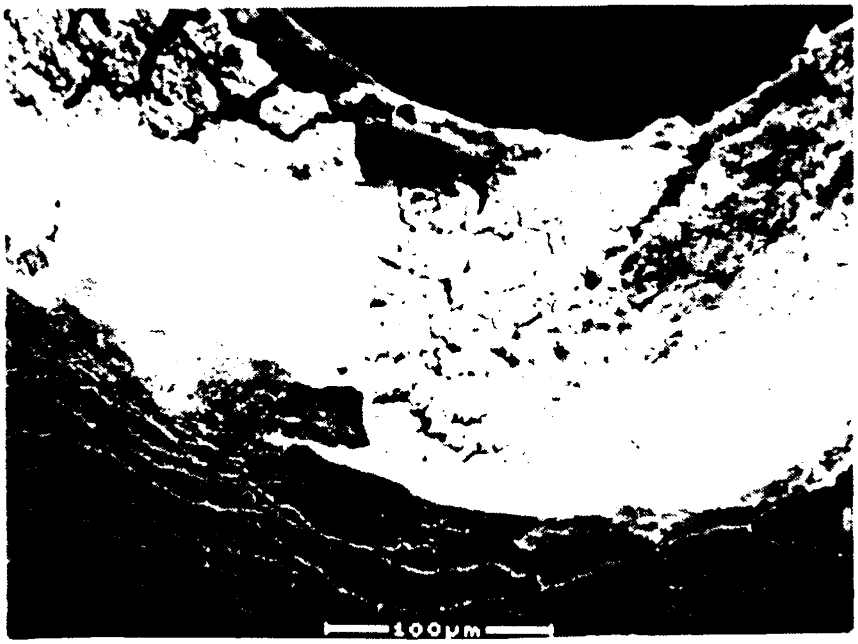
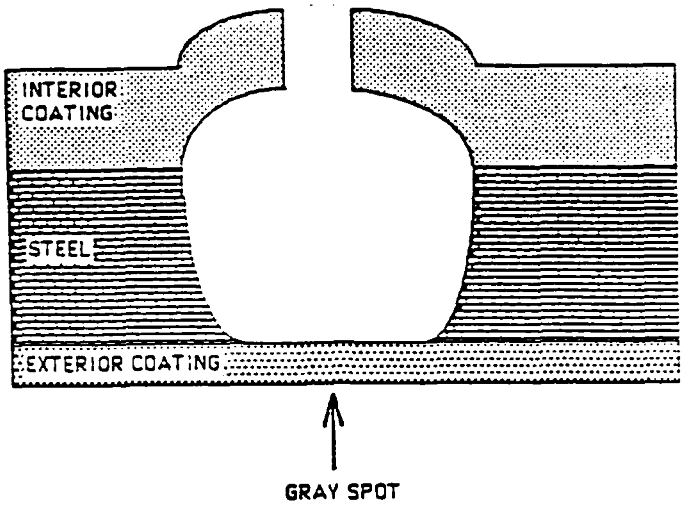


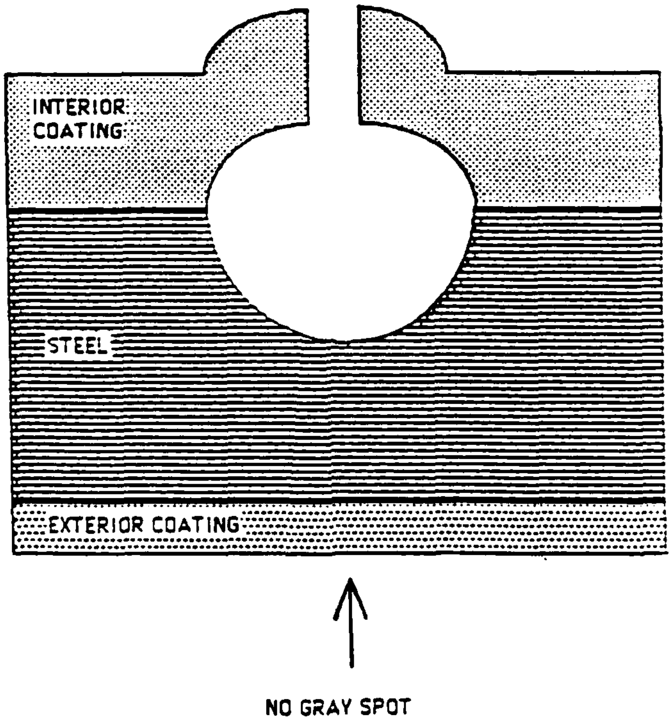
Figure 9. Detailed view of a pit wall after ultrasonic cleaning (T134).

Figure 10. Microcracks in the epoxy layer at the stressed area in the interior of a virgin tray after vinyl topcoat removal.

A review of our findings clearly shows that leakers are a consequence of corrosion which initiates in the interior of the tray pack at the coating/steel interface and proceeds outward to the exterior surface where either a gray spot or pin hole is observed. All of the food stuffs contain water and salt, an aggressive environment. Chloride ions and H_2O penetrate into the vinyl coatings followed by localized mass transport of ions through coating defects. This process is continued into and through the epoxy coating at pores or cracks with follow-on substrate (steel) ionic/electronic charge transfer reactions (corrosion) occurring at the coating/metal interface. Corrosion beneath an organic coating is a special example of an occluded corrosion cell where a pocket of liquid is isolated from the bulk liquid (as in pitting, crevice corrosion, or stress corrosion cracking) and chemical changes occur in the liquid resulting in intensification of localized corrosion. Until corrosion (pitting) proceeds through the entire steel thickness, there is no evidence of a gray spot at the exterior clear epoxy/metal interface. This difference is shown schematically in Figures 11a and 11b.



(a) Interior



(b) Exterior

Figure 11. Schematic models showing the formation of blister/pit on the interior with 'gray spots' or no 'gray spots' on the exterior of the tray.

Assessment of Corrosion Resistance of Coatings by AC Impedance Measurements

Electrochemical impedance techniques are finding increased application in both corrosion research and the evaluation of the performance of organic coating/metal systems. Since the corrosion processes occurring on metal substrates under organic coatings are electrochemical in nature, assessments of the corrosion resistance of organic coatings have been made employing electrochemical measurements, and much data has been reported showing that various electrical parameters are important in selecting a corrosion resistant organic coating. Leidheiser[1] has reviewed a number of electrochemical and electrical measurement techniques for predicting corrosion at the metal/organic coating interface, he reported that a coating system resistance measured by either AC or DC resistance techniques degraded with time and a lower limit of about 10^6 ohms/cm^2 existed, below which corrosion occurred underneath the coating. Leidheiser associated this resistance degradation with ion and water penetration into the coating, with current transport of ions through the coating, and then follow-on electrochemical reactions at the coating/metal interface. Mikhailovskii[2] has reported that in many cases the DC resistance may not be a true measure of the corrosion resistance of paints. Mansfeld[3] has reviewed the current status of polarization resistance measurement, and points out the advantages of the AC Impedance Technique in obtaining the polarization resistance especially for measurements on low conductivity media and for systems with low corrosion rates. Mansfeld has also reviewed methods of analyses of AC impedance data.[4,5] Levy et al.[6] have obtained values of polarization resistance (Rp) for epoxy primers as a means of assessing their ability to protect magnesium from corrosion in chloride environments using both Nyquist and Bode plots.

Scantlebury et al.[7] have applied analysis of Nyquist plots (also known as Cole-Cole) to provide an estimate of the film integrity and protective capacity of chlorinated rubber and coal tar epoxy coatings.

AC impedance measurements were carried out to evaluate the performance of the tray pack coatings/metal systems. A PARC 273 potentiostat was employed in conjunction with a PARC 5208 Lock-in Analyzer, Apple IIe computer, and the PARC Softcorr 368 program. Measurements were obtained at the corrosion potential over the frequency range 100K Hz - 0.01 Hz for exposure times up to 28 days in 3.5% NaCl solution at pH 5.

Figure 12 contains Bode plots, (a) log $|Z|$ versus log frequency and (b) θ phase angle (deg) versus log frequency, which compare the behavior of the new HGW coating exposed to 3.5% NaCl solution for 14 and 28 days. The polarization resistance, determined by extrapolation from the linear region at low frequencies to the log $|Z|$ axis intercept, (see Figure 12a) remains relatively unchanged from 0 to after 14 and 28 days of exposure. However, the phase angle plot (see Figure 12b) shows significant differences in behavior as a function of exposure time. The new peak after 14 days indicates another phenomena is occurring, differing from the initial one of penetration of the solution into the coating. This probably represents corrosion at the metal/coating interface.

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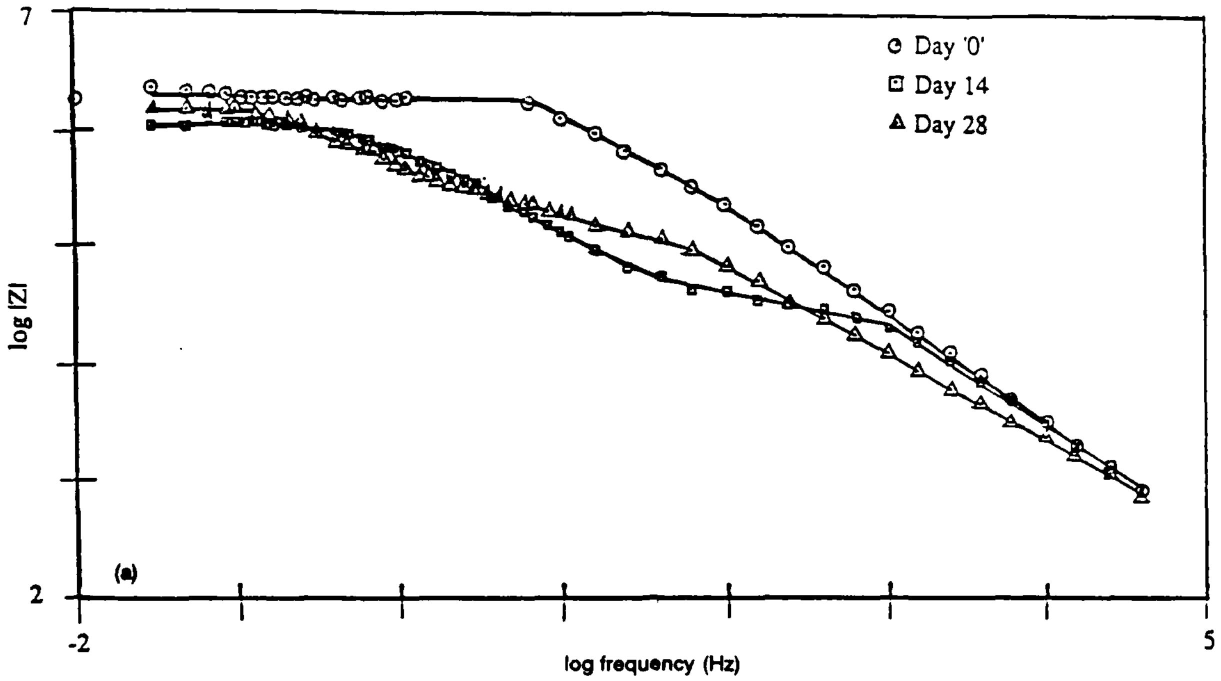

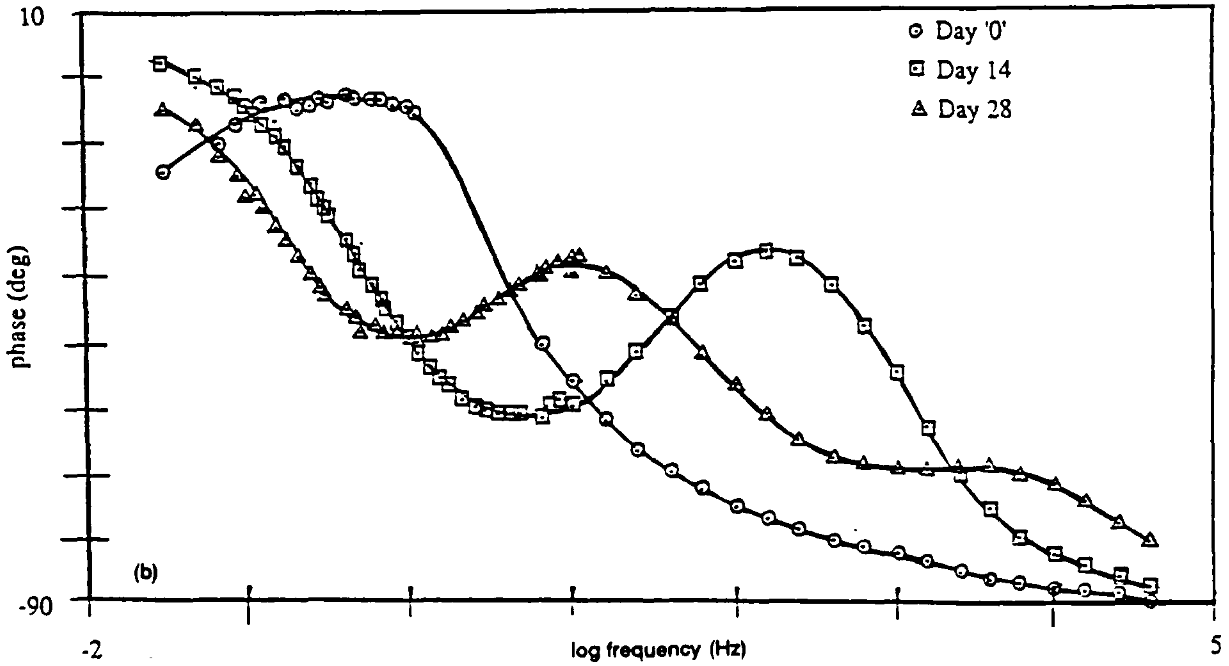

Figure 12. Bode plots for (a) magnitude and (b) phase angle for HGW coating tested in 3.5% NaCl for 0, 14 and 28 days exposure.

Figure 13 contains Nyquist plots for the same coating exposed for 0, 14, and 28 days to 3.5% NaCl solution. The response curve after one day of exposure is a relatively large semi-circle. The impedance behavior after 14 and 28 days shows that the high frequency intercept of the curves with the Z'-axis is the same, but the response curves become smaller semi-circles (decreasing in area with increased exposure time) with the center of the semi-circle lying along the Z'-axis. An estimate of the polarization resistance may be made from the intersection of the low frequency end of the semi-circle with the Z'-axis. Thus, the polarization resistance decreases with increasing exposure time.

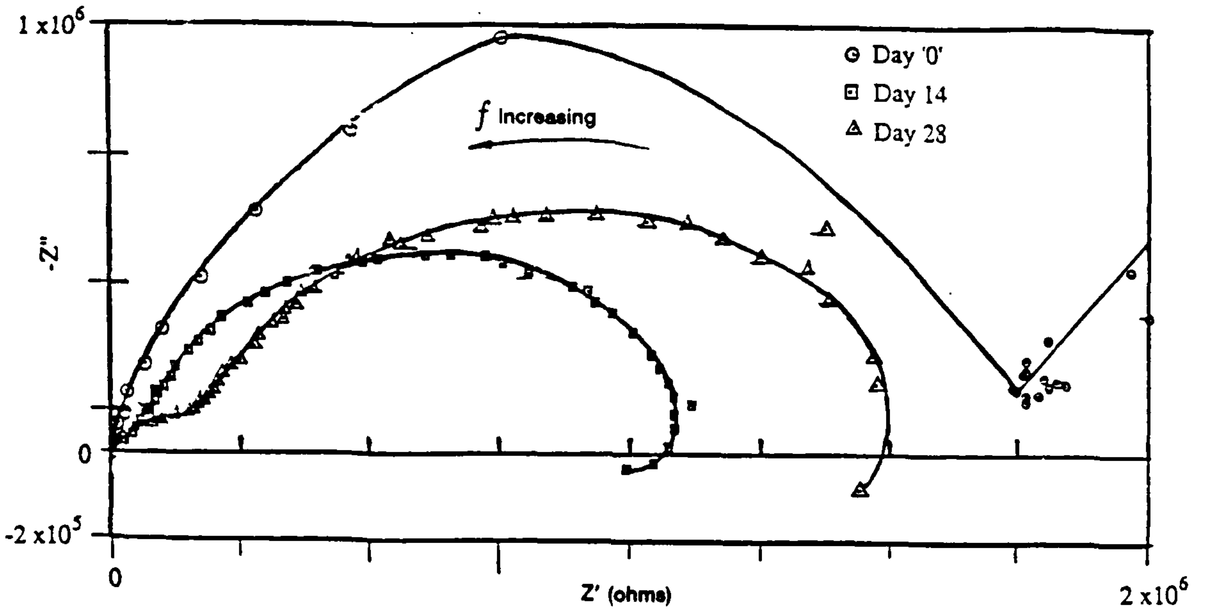

Figure 13. Nyquist plot for the same HGW coating, as shown in Figure 12.

Similar AC impedance measurements in 3.5.% NaCl solution were made on the current coating system. Bode and Nyquist plots for this system are contained in Figures 14a, 14b, and 15, respectively; generally, the impedance behavior of the multilayer system is qualitatively similar to that of the new high gloss (HGW) coating. Nevertheless, there are some discrete differences which are discernable from the plot of polarization resistance as a function of exposure time, as shown in Figure 16. The presently used coating system has a much higher initial polarization resistance (Rp) than the HGW coating (1×10^{10} ohms cm^2 versus 6.3×10^6 ohms cm^2), but as the coating system is exposed to 3.5% NaCl solution, Rp decreases with exposure time and levels off to 6.3×10^7 ohms cm^2. Although some coating degradation has occurred, Rp after 28 days of exposure remains significantly above the critical value[1] of 10^6 ohms cm^2, below which corrosion of the tin-free steel occurs. The Rp of the HGW coating is consistently much lower and even approaches the critical value of 10^6 ohms cm^2 after exposure time of 28 days.

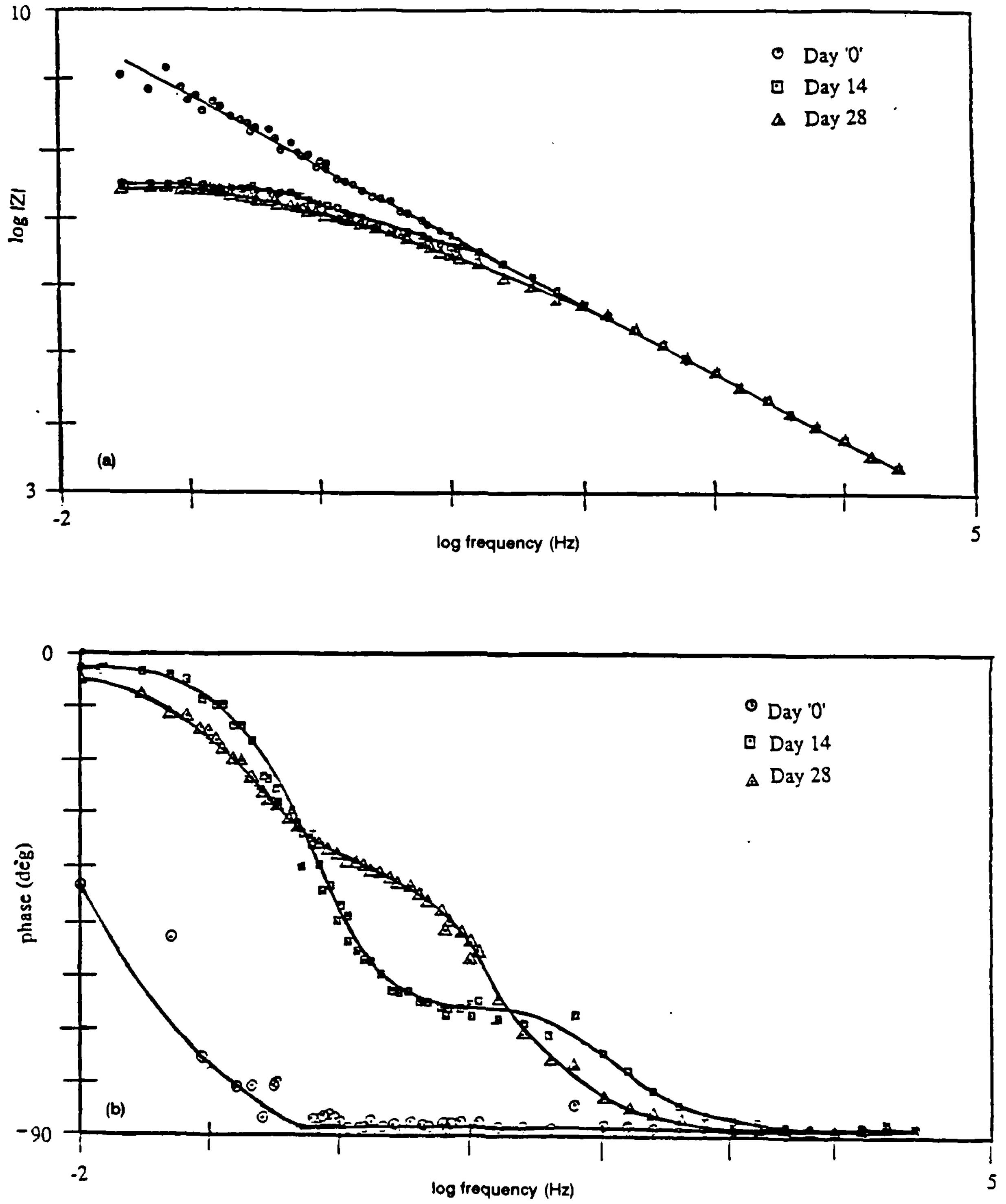

Figure 14. Bode plots for (a) magnitude and (b) phase angle for the current coating system tested in 3.5 % NaCl for 0, 14 and 28 day exposures.

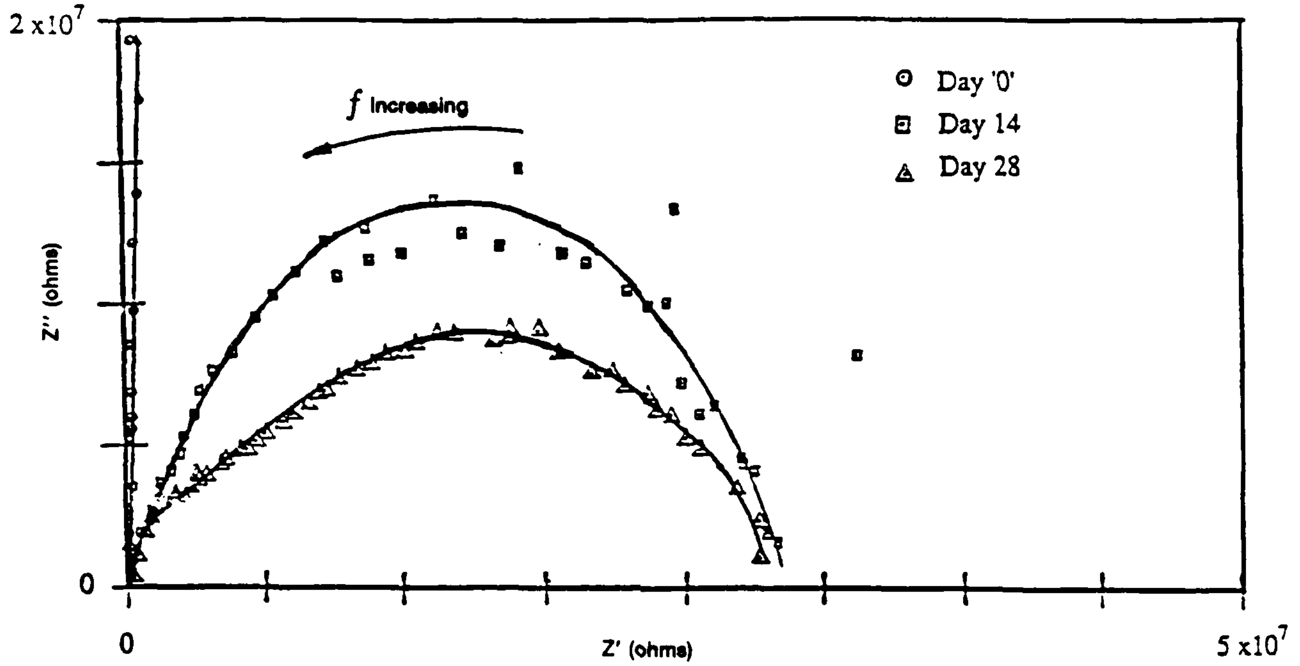

Figure 15. Nyquist plot for the current coating system data as shown in Figure 14.

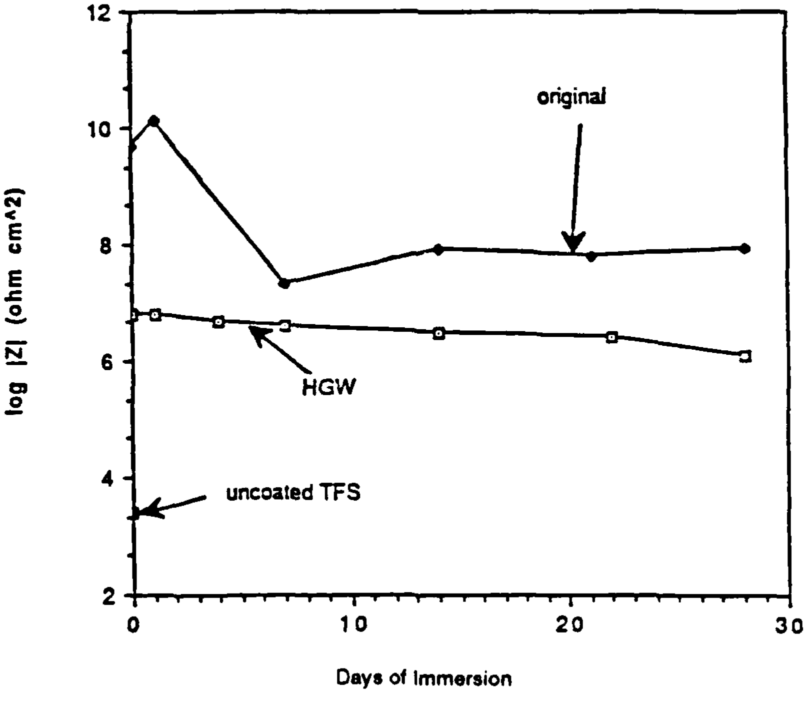

Figure 16. Comparison of the polarization resistance of the HGW and the current coating system as a function of days of exposure to 3.5% NaCl.

CONCLUSIONS

1. Accelerated corrosion tests exposed defects in the interior coatings which led to corrosion of the tin-free steel within one to several days of exposure in a NaCl environment.

2. The presence of "gray spots" on the exterior surface of the tray pack could be related to an existing defect in the directly opposite surface of the interior coating.

3. Microscopic examination in conjunction with AC impedance measurements have shown that "gray spots" are the consequence of the following sequential steps leading to corrosion of the tin-free steel.

(a) Water and ions penetrate the interior coatings through localized microscopic defects such as pores or cracks.

(b) Conductive paths develop in the coatings in which there is a low resistance pathway between the tin-free steel and the bulk electrolyte (liquid).

(c) Once sufficient electrolyte has penetrated the coating, an anodic reaction takes place.

(d) This reaction intensifies (causing pitting) in tin-free steel as a result of the presence of an occluded cell.

(e) Once the pit has penetrated through the entire thickness of the tin-free steel, a gray spot appears under the exterior clear epoxy coating.

4. The greater values of polarization resistance (Rp) (extrapolated from AC impedance measurements) for the current multiphase coating system, indicated that the present coating has a higher degree of corrosion resistance than the new single phase HGW coating. Thus, Rp measurements can be used as a qualitative means for accelerating pitting corrosion testing in tray packs. More work to evaluate other coating systems would be warranted.

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METALLURGICAL ANALYSIS OF LEAKING
TRAY PACKS - Kuan S. Lei, F. Chang, and
Milton Levy

Technical Report MTL TR 90-30, May 1990, 21 pp-
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Key Words

Tin-free steel (TFS)
Organic coatings
Corrosion resistance

A metallurgical analysis was performed to determine the cause of leaking tray packs and the origin and development of "gray spots" which represent potential leak sites. Tray packs are fabricated of tin-free steel (TFS) and are protected with multilayered coatings on both interior and exterior surfaces. Accelerated corrosion tests were carried out in order to monitor the progress of corrosion in a simulated long-term exposure environment. These tests exposed defects in the interior coatings (pores, cracks, blisters) which led to pitting corrosion of the TFS within a few days of exposure to the chloride environment. The presence of "gray spots" on the exterior surface of the tray packs was related to an existing defect in the directly opposite surface of the interior coatings. Microscopic examination, in conjunction with AC impedance measurements, showed that water and aggressive ions contained in the food penetrate into and through interior coatings, through localized defects such as pores and cracks, causing pitting of the TFS which intensifies as a result of the presence of an occluded electrochemical cell at the coating/metal interface. Once the pit has penetrated through the entire thickness of the TFS, a gray spot appears under the exterior clear epoxy coating. AC impedance measurements also were made to compare the corrosion resistance of the current multilayered coating system with a newly developed single phase coating (HGW). The greater values of polarization resistance derived from these measurements indicated that the current multiphase coating system has a higher degree of corrosion resistance than the new HGW coating.

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