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Microbiologically influenced corrosion (MIC) has received increasing attention from engineers, materials scientists, and corrosion specialists. In field and laboratory studies, basic surface analytical examinations must be correlated to knowledge of the overall corroding system in order to conclude the presence of MIC. Preliminary observations and microbiological, chemical, microscopic, and metallurgical techniques are discussed.

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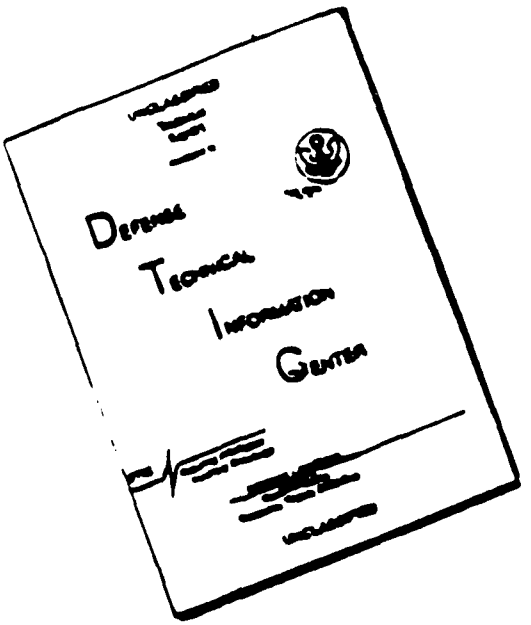

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Surface Analytical Techniques for Microbiologically Influenced Corrosion—A Review

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ABSTRACT: Microbiologically influenced corrosion (MIC) has received increasing attention from engineers, materials scientists, and corrosion specialists. In field and laboratory studies, basic surface analytical examinations must be correlated to knowledge of the overall corroding system in order to conclude the presence of MIC. Preliminary observations and microbiological, chemical, microscopic, and metallurgical techniques are discussed.

KEYWORDS: microbiologically influenced corrosion (MIC), biofilms, surface analysis

Microorganisms attach to all engineering materials in contact with natural waters and colonize surfaces to produce biofilms. The biofilms are varied in composition but usually include bacteria, algae, and fungi, in addition to exopolymeric material that provides attachment and structural integrity. A large fraction of the biofilm is adsorbed and entrapped materials such as solutes, heavy metals, and inorganic particulates, in addition to cellular constituents [*1*]. Cells within biofilms grow, reproduce, and form colonies that are physical anomalies on a metal surface; local anodes and cathodes and differential aeration cells result (Fig. 1). Under aerobic conditions, areas under respiring colonies can become anodic and surrounding areas cathodic. A thick biofilm can prevent diffusion of oxygen to cathodic sites and diffusion of aggressive anions, such as chloride, to anodic sites. Outward diffusion of metabolites and corrosion products is also impeded. If areas within the biofilm become anaerobic, the cathodic mechanism can change to reduction of water or microbiologically produced H_2S.

Biofilms can be either beneficial or detrimental in industrial processes. They remove dissolved and particulate contaminants in fixed film biological systems, such as trickling filters, rotating biological contactors, and fluidized bed wastewater treatment plants. Biofilms can determine water quality by influencing dissolved oxygen content and by serving as a sink for toxic and/or hazardous materials. Microorganisms within biofilms can be used to recover minerals and to degrade hydrocarbons [*2*]. However, biofilms form undesirable deposits on engineering surfaces causing reduced heat transfer [*3*], increased fluid frictional resistance [*3*], plugging [*4*], and corrosion [*4*].

The term microbiologically influenced corrosion (MIC) is used to designate corrosion resulting from the presence and activities of microorganisms within biofilms on a material

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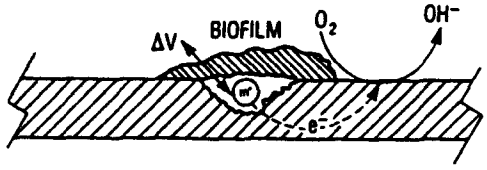

FIG. 1—*Differential aeration cell resulting from microbial colony on metal surface.*

surface. Microorganisms can accelerate and control corrosion reactions by several mechanisms: formation of differential or concentration cells, formation of aggressive metabolites, such as sulfides and organic and inorganic acids; metal oxidation and reduction, and deactivation of corrosion inhibitors. Iron-oxidizing, sulfur-oxidizing, iron-reducing, sulfate-reducing, acid-producing, slime-producing, ammonia-producing, and hydrogen-producing bacteria have been implicated in the corrosion of metals and alloys. Sulfate-reducing bacteria (SRB) are commonly found to be responsible for MIC in anaerobic environments through the production of H_2S. Metal-depositing bacteria, especially iron-oxidizing genera, form dense deposits of cells and metal ions, creating oxygen concentration cells and under-deposit corrosion. Acidic bacterial exopolymers can bind metal ions from the aqueous phase, increasing corrosion rates by providing an additional cathodic reaction.

MIC has received increased attention by corrosion scientists and engineers in recent years with the development of surface analytical and electrochemical techniques that can quantify the impact of microbes on electrochemical phenomena and provide details of corrosion mechanisms. MIC has been documented for metals exposed to seawater, fresh water, demineralized water, process chemicals, food stuffs, soils, aircraft fuels, human plasma, and sewage. The chemical process, oil and gas, and power generation industries and the U.S. military have acknowledged the occurrence and prevalence of MIC in their operating systems. In the past ten years there have been at least 20 international conferences that included sessions on the subject.

Investigations for MIC can usually determine only if conditions are appropriate for MIC. Experience has shown that MIC is considered only when other forms of nonbiological corrosion have been eliminated. This paper will review field and laboratory surface analytical procedures for investigating a corroding system to determine if MIC may be a causative agent. Some are applicable to field and laboratory use, while others are only useful for research. Related electrochemical techniques used to identify mechanisms and monitor and quantify electrochemical parameters and corrosion rates have been discussed elsewhere [5].

Preliminary Examination of a Corrosion System

The Corroding Sample

1. Metal Composition—Metals listed as commercially pure actually contain a variety of impurities and imperfections that influence corrosion. In general, as purity increases, the tendency for a metal to corrode is reduced. However, high purity metals frequently have low mechanical strength, leading to the use of alloying elements to improve mechanical, physical, fabrication, and corrosion characteristics [6]. Alloy composition, manufacturing specifications such as surface finish and heat treatments, and presence of protective coating influence susceptibility to MIC [7].

2. Macroscopic Examination—Color photographs of the corroded material while still wet and before extensive handling can be invaluable for reference and documentation.

a. *Visible fouling*

Extensive biological foul
presence of algae, in additic
oxygen (photosynthesis) tha
consume oxygen (respiratio
photosynthetic biofilm may
stainless steel so that it ap
(Fig. 2). Algae and fungi m
al. have shown that there
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b. *Localized corrosion*

(1) Form—MIC is loc
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stainless steel. When SRB ar
is often found under adjacer
stringers in weld areas of s
graphitization where the cor
nickel and cupronickel alloys
rings [12].

(2) Location—Distribu
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of low flow such as in bends
are common sites for MIC, e
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(3) Material within Pits—
found in open pits in galvan
and anaerobic SRB [13].

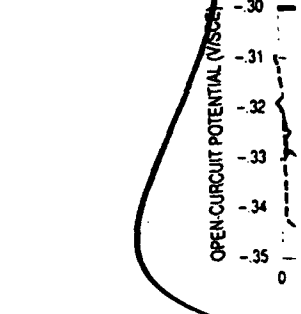

FIG. 2—*Open-circuit potent*
biofilm under a 6h light/dark r
slot [8].

a. *Visible fouling*

Extensive biological fouling with filamentous material, slime, and debris suggests the presence of algae, in addition to fungi and bacteria. In the presence of light, algae produce oxygen (photosynthesis) that can accumulate in the biofilm. In the absence of light, algae consume oxygen (respiration) and reverse the process. Dowling et al. [*8*] showed that a photosynthetic biofilm may influence ennoblement of the open circuit potential of type 316L stainless steel so that it approaches the potential above which pits can initiate and grow (Fig. 2). Algae and fungi may also produce aggressive metabolites. Additionally, Little et al. have shown that there is no correlation between the thickness of the biofilm and the chemistry at the biofilm/metal interface [*9*]. Localized cells of pH values 5.2-9.2 were measured randomly at depths within an estuarine biofilm on type 304 stainless steel.

b. *Localized corrosion*

(1) Forms—MIC is localized corrosion and can appear as pitting, crevice corrosion, under-deposit corrosion, dealloying, or stress corrosion cracking. The form, shape, and depth of the corroded areas should be noted. Pits associated with MIC often have a small surface opening with a larger subsurface cavity. SRB produce open pitting or gouging on stainless steel. When SRB are active along edges of gasketed joints, shallow crevice corrosion is often found under adjacent gaskets. Subsurface tunneling has been observed along ferrite stringers in weld areas of stainless steel [*10*]. SRB attack on cast iron typically produces graphitization where the corroded areas are filled with a soft skeleton of graphite [*11*]. On nickel and cupronickel alloys, SRB are reported to produce conical pits containing concentric rings [*12*].

(2) Location—Distribution of corrosion within the sample is important. Frequently, pitting of stainless steels is located in the heat-affected zone, fusion line, and adjacent base metal of welds [*10*]. Kobrin described pitting attack in weld seams of a type 316L stainless steel storage tank after hydrotesting due to metal-depositing bacteria (Fig. 3) [*12*]. Regions of low flow such as in bends, elbows, or crevices due to engineering design and fabrication are common sites for MIC, especially where low oxygen concentrations encourage anaerobic bacterial growth. Any recurring directional pattern of localized attack may be related to turbulence or impinging flow.

(3) Material within Pits—Tatnall reviewed a case history where fine dark particles were found in open pits in galvanized steel, identified to include aerobic sulfur-oxidizing bacteria and anaerobic SRB [*13*].

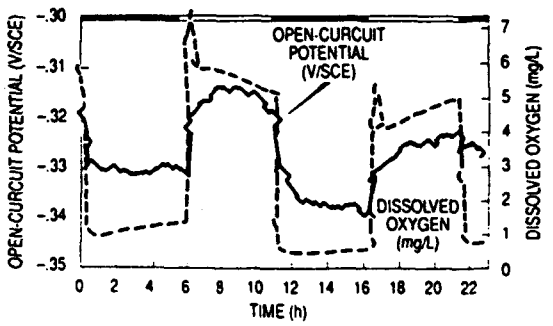

FIG. 2—*Open-circuit potential and dissolved oxygen oscillations associated with an* Anabena *sp. biofilm under a 6h light/dark regime. Light period denoted by white slot, dark period denoted by black slot* [*8*].

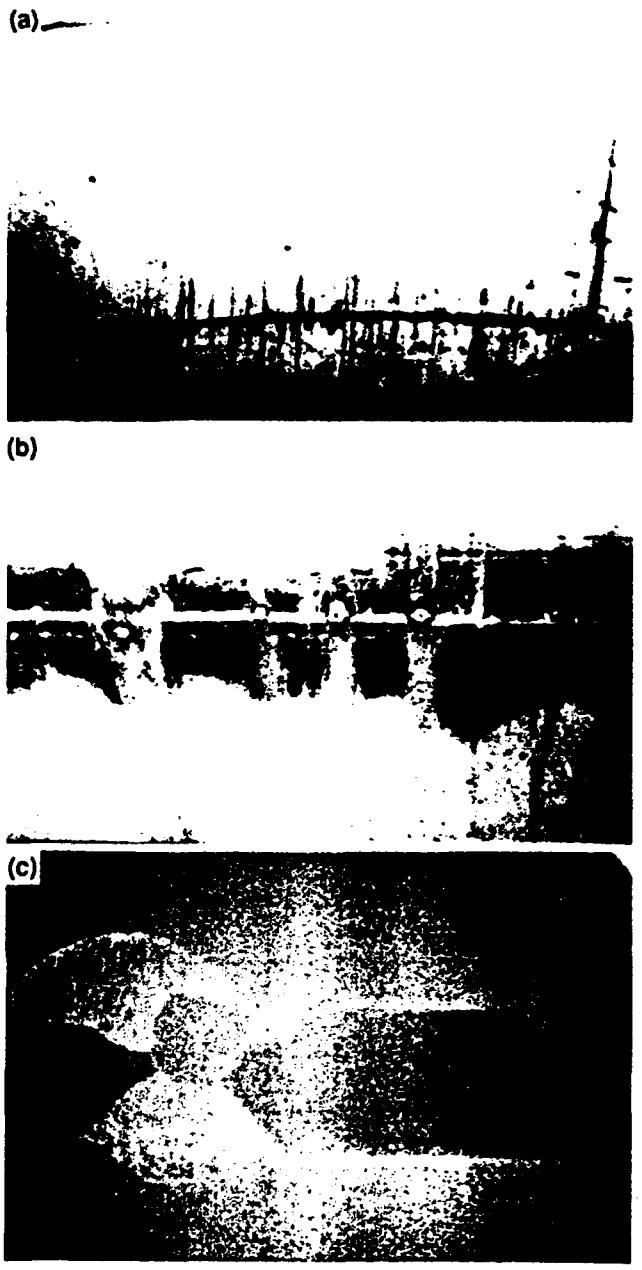

FIG. 3—*Tubercle formation on stainless steel [12]: (a) = rust colored streaks normal to weld seams in sidewall of 316L tank formed after 1 month exposure to stagnant hydrotest water, (b) = wet deposit of metal-depositing bacteria, and (c) = cross-section through pitted weld seam.*

c. *Corrosion products*

Discrete mounds or columns (tubercles) can develop on metal surfaces as a result of microbial activities. Morphology and location are often indicative of the causative microbial species. Deposit shape, color, and texture should be noted. SRB produce characteristic black deposits of FeS on steel and stainless steels. Distinctive reddish-brown, hemispherical, or conical tubercles with a small "chimney shape" near the center on the surface of steel and subsurface pitting are characteristics of iron bacteria activity [*14*].

Bacterial deposits usually have a soft slimy texture when fresh and wet. In the presence of slime-formers, deposits are more irregular and may appear layered. In anaerobic conditions where SRB involvement is suspected, the deposit may be screened for the presence of H_2S by odor and testing with HCl [*11*].

Environment of the Corroding System

In an ideal investigation of MIC, the corrosion environment would be available for inspection. Factors of interest include the following:

(1) Presence, Absence, Cycles of Light—This would influence biofilm composition, respiration, and metabolic activities [*8*].

(2) Aqueous Medium—Microorganisms within the biofilm are capable of maintaining an environment that is radically different from that of the bulk medium in terms of pH, dissolved oxygen, and organic and inorganic species [*9*]. Interfacial chemistry cannot be predicted from measurement of any set of parameters in the bulk medium. Similarly, the numbers and types of bacteria within biofilms cannot be predicted or determined by measuring planktonic microorganisms [*15*]. The following parameters for the aqueous medium offer supportive data for investigation of MIC and causative organisms.

a. *Temperature*—Microorganisms have been found at water temperatures from 1°C in Antarctic waters [*16*] to 320°C in deep-sea hydrothermal vents [*17*].

b. *Salinity*—Bacteria are commonly found in fresh and open ocean waters.

c. *Dissolved oxygen*—Bacteria are found in 0 to 100% O_2 concentrations.

d. *Water chemistries*—Including organic carbon nutrients, NO_3, CO_2, O_2, SO_4, and other compounds that may serve as terminal electron acceptors in respiratory metabolism. The presence and concentration of nitrites, phosphates, and sulfides; ionic materials such as chlorine, sulfur and phosphorous; metals, and acids is important. For example, breakdown of the protective passive oxide film on stainless steel occurs in the presence of the chlorides.

e. *Water microbiology*—See microbiology discussion.

f. *Direction and velocity of flow*—Hydrodynamic shear stress, related to flow, influences transport, transfer, and reaction rates within the biofilm, as well as biofilm detachment.

(3) System Relationship to Nearby Upstream Industry—Environmental ground or air pollution and terrestrial influence, as compared to open ocean.

(4) Operating History of Corroding System—As an example, there have been several documented cases of MIC in the nuclear industry where shutdown after hydrotesting with natural waters has resulted in extensive pitting failures [*18*]. Inadequate drainage left stagnant areas conducive to bacterial attack. Antifouling and cathodic protection measures in use should be known.

l streaks ormal to weld seams rotest wa r. (b) = wet deposit l seam.

Collection and Transport of Corroding Sample and Medium

Pope [*11*], Tatnall [*14*], and Stoecker [*19*] have described sample collection for the study of MIC. Swabs should be obtained from the base metal and within pits beneath tubercles. Samples of tubercular material and aqueous medium, in addition to any other items in the environment of interest should be collected. General recommendations are to collect and analyze intact specimens, maintained in natural liquid medium, as soon as possible after disturbing the normal operating system. Samples should be taken in clean sterile containers and chilled until examination within 12-24 h. Specimens to be studied microscopically should be fixed in preservatives such as 2-4% formaldehyde or glutaraldehyde to maintain structural integrity.

Microbiology

It must be remembered that biofilms are a total community with synergistic relationships between organisms, producing activities different than those from isolated species. Cultures only provide identification of species present.

Standard microbiological practices for general and selective cultures are commonly described. General plate counts may be misleading because results do not necessarily correlate with bacteria directly related to MIC. Using knowledge of the corroding system, including oxygen content, metal alloy involved, and other parameters, a microbiologist could determine investigative directions such as using Postgate medium [*20*] where SRB are suspected.

Commercially prepared media and test kits are available for on-site and laboratory screening. Little et al. have described several for the detection of SRB [*21*].

Culture Techniques

The American Petroleum Institute (API, New York, NY) Recommended Practice (RP 38) [*22*] for the enumeration of SRB in subsurface injection waters specifies sodium lactate as the carbon source. When bacteria are present in the sample, they reduce sulfate in the medium to sulfide that reacts with iron in solution to produce black ferrous sulfide. Blackening of the medium over a 28-day period signals the presence of SRB. A solid medium technique termed "agar deeps" uses a modification of API with sodium sulfite as the reducing agent/oxygen scavenger [*23*]. An agar slant is inoculated, oxygen is excluded, tube is sealed, incubated for 5 days, and observed for blackening.

Direct Methods

Unlike culturing techniques, direct methods for detecting and quantifying SRB do not require SRB growth. Instead, direct methods measure constitutive properties including: adenosine-5'-phosphosulfate (APS) reductase [*23*], hydrogenase [*24*], cell-bound antibodies [*25*], and DNA [*26*]. Attempts have also been made to use adenosine triphosphate (ATP) [*27*] and radiorespirometric measurements for estimates of SRB activity [*28*].

The APS reductase antibody method was developed by Tatnall [*23*]. APS reductase is an intercellular enzyme found in all SRB. Briefly, cells are washed to remove interfering chemicals including hydrogen sulfide and lysed to release APS reductase. The lysed sample is washed and exposed to a color-developing solution. In the presence of APS reductase a blue color appears within 10 min. The degree of color is proportional to the amount of enzyme and roughly to the number of cells from which the enzyme was extracted. Similarly, a procedure has been developed to quantify hydrogenase from SRB that requires that cells

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Field and laboratory epifl have been developed by D. H detection of specific antibodie antibody produced in goats, the SRB cells. In the laborat that enables bacterial cells m fluorescence microscope. In t enzyme (alkaline phosphatase a visible color proportional to

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ATP assays estimate the to ATP in a sample. ATP is a ATP assay techniques may be of SRB [*27*]. The procedure r salts that may interfere with th cell ATP. An enzyme then reac light can be measured with a p the total light emitted.

Microscopy

Because MIC cannot be ver corrosion products, it is esse established between microorg techniques have been used to Epifluorescence microscopy h surfaces [*25*]. This technique distinguish individual cells wi croscopy and it is sometimes i mission electron microscopy (T throughout corrosion layers [and thin sectioning. Tradition dispersive spectroscopy (EDS roded areas and to determine of the base metal, pit area, c Presence, morphology, and dis ence of polymeric material m distribution of bacteria in cor year of service [*30*].

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be concentrated by filtration from water samples [24]. Solids, including corrosion products and sludge, can be used without pretreatment. The sample is exposed to an enzyme extracting solution for 15 min and placed in an anaerobic chamber from which oxygen is removed by hydrogen. The enzyme reacts with excess hydrogen and simultaneously reduces an indicator dye in solution. The activity of the hydrogenase is established by the development of a blue color within 4 h. Color intensity is proportional to rate of hydrogen uptake.

Field and laboratory epifluorescence cell surface antibody methods for detecting SRB have been developed by D. H. Pope [25]. Both methods are based on the use and subsequent detection of specific antibodies, produced in rabbits, that react with SRB cells. A secondary antibody, produced in goats, is then reacted with the primary rabbit antibodies bound to the SRB cells. In the laboratory method, the goat antibodies are linked to a fluorochrome that enables bacterial cells marked with the secondary antibody to be viewed with an epifluorescence microscope. In the field method, the goat antibodies are conjugated with an enzyme (alkaline phosphatase) that can then be reacted with a colorless substrate to produce a visible color proportional to the quantity of SRB present.

Hogan has described a nonisotopic, semiquantitative procedure for the detection of *Desulfobacterium* and *Desulfotomaculum* using DNA probes that are labeled with an acridinium ester and is sensitive to 10^2 organisms/mL [26]. DNA probes are directed towards ribosomal RNA and may be viewed as consisting of three to four steps: (1) sample handling, (2) binding the probe to the target, (3) removal or destruction of the unbound probe, and (4) detection and quantification of the reporter group on the bound probe.

ATP assays estimate the total number of viable organisms by measuring the amount of ATP in a sample. ATP is a compound found in all living matter. Littman proposed that ATP assay techniques may be used with oilfield water samples to estimate relative numbers of SRB [27]. The procedure requires that a water sample be filtered to remove solids and salts that may interfere with the test. The filtered sample is added to a reagent that releases cell ATP. An enzyme then reacts with the ATP to produce a photochemical reaction. Emitted light can be measured with a photometer and the number of bacterial cells is estimated from the total light emitted.

Microscopy

Because MIC cannot be verified by morphology of localized corrosion or composition of corrosion products, it is essential in the diagnosis of MIC that a spatial relationship be established between microorganisms, substratum metal, and corrosion. Several microscopic techniques have been used to document numbers and types of microorganisms on surfaces. Epifluorescence microscopy has been used to evaluate the distribution of cells on corroded surfaces [25]. This technique requires sample fixation and staining. It is often difficult to distinguish individual cells within a densely populated biofilm using epifluorescence microscopy and it is sometimes impossible to penetrate corrosion products with stains. Transmission electron microscopy (TEM) has been used to demonstrate microbial cells distributed throughout corrosion layers [29]. TEM requires sample fixation, dehydration, embedding and thin sectioning. Traditional scanning electron microscopy (SEM), coupled with energy dispersive spectroscopy (EDS), has been used extensively to demonstrate bacteria in corroded areas and to determine surface chemistries resulting from MIC. Elemental chemistry of the base metal, pit area, corrosion products, and general biofilm should be identified. Presence, morphology, and distribution of microorganisms within the biofilm, and the presence of polymeric material must be determined. Figure 4 shows localized corrosion and distribution of bacteria in corrosion products from a copper/nickel piping system after 1 year of service [30].

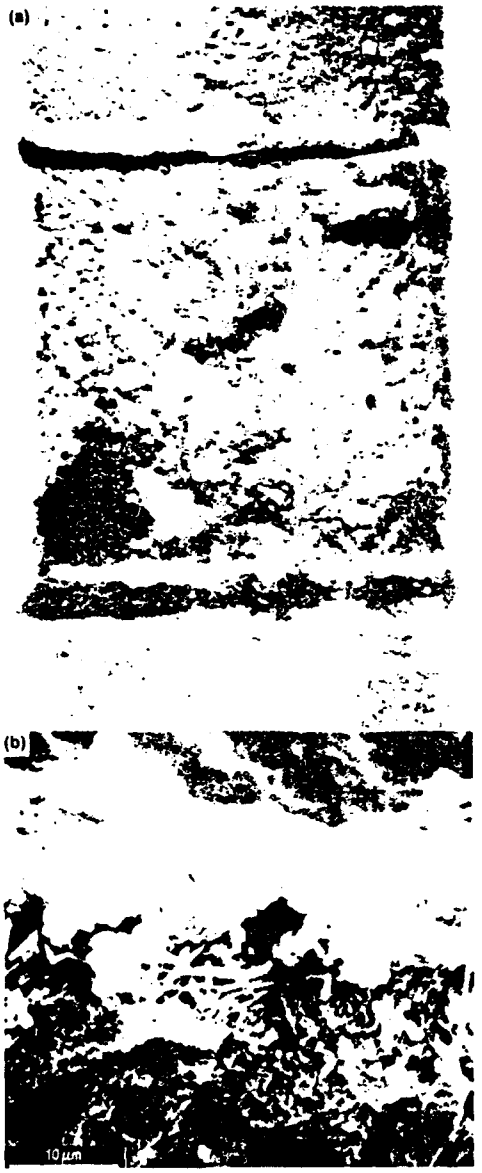

FIG. 4—*Pitting in copper/nickel piping system after 1 year in service [30]: (a) = pitted area and (b) = bacteria in cross-section of pitted area.*

Preparation of biological material for SEM requires extensive manipulation, including fixation, dehydration, and either air or critical-point drying because the SEM operates at high vacuum. Nonconducting samples, including biofilms, must be coated with a conductive film of metal before the specimen can be imaged. Uncoated nonconductors build up local concentrations of electrons, referred to as "charging," that prevent the formation of usuable images. EDS can be used to determine the elemental composition of surface films in the

SEM, but EDS analyses must
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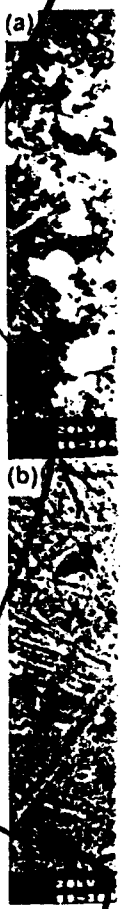

FIG. 5—*ESEM images com*
as taken directly from water and

SEM, but EDS analyses must be completed prior to deposition of a thin metal coating. EDS data are typically collected from an area, the specimen removed from the specimen chamber and coated with a conductive layer, and returned to the SEM. The operator attempts to relocate and photograph the precise area from which the EDS data were collected. Little et al. [*31*] demonstrated that sample preparation for SEM, including the solvent removal of water and air or critical point drying, decreases areal coverage of the surface by the biofilm, removes cells from the biofilm, removes extracellular polymeric material that may contribute to corrosion, and decreases the concentration of metals bound within the matrix of the exopolymer (Fig. 5).

Environmental scanning electron microscopy (ESEM) was used to demonstrate that the number and types of microorganisms on copper surfaces have been underestimated by SEM. ESEM provides fast, accurate images of a biofilm (Fig. 6), its spatial relationship to a corrosion site, as well as surface chemistry, without extensive manipulation of the sample. This instrument uses a unique secondary electron detector capable of forming high resolution images at pressures in the range of 0.1 to 20 torr. At these relatively high pressures, specimen

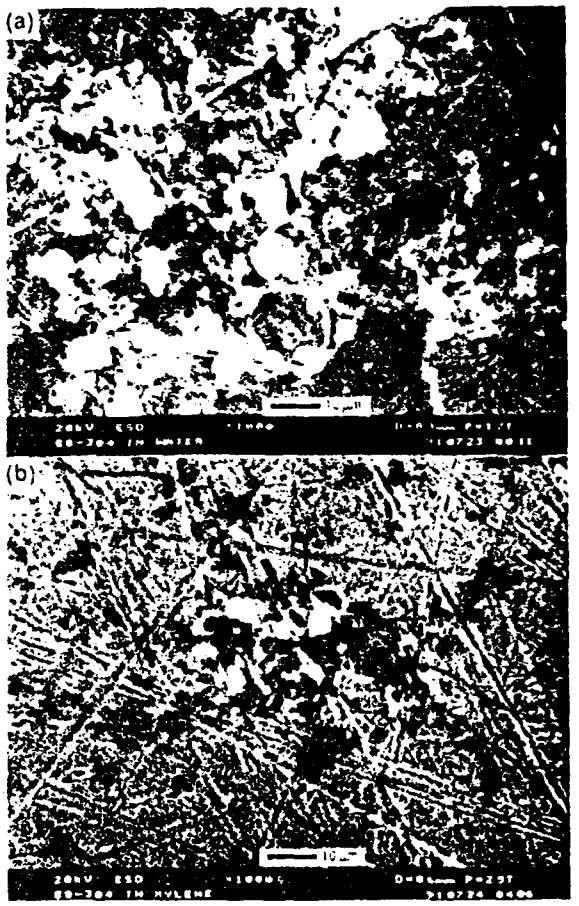

FIG. 5—*ESEM images comparing biofilm coverage when wet and after removal of water* [*31*]: (*a*) = *as taken directly from water and* (*b*) = *after acetone-xylene removal of water.*

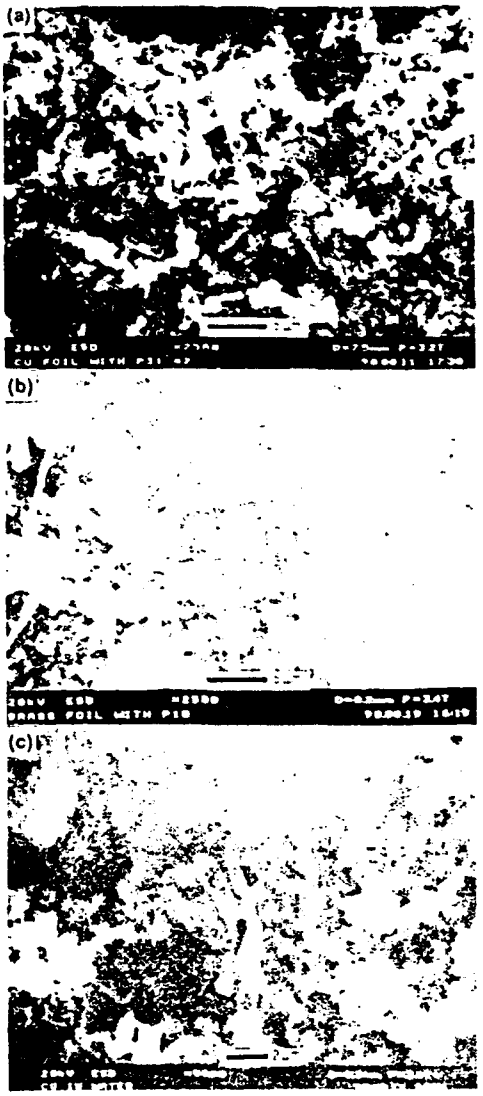

FIG. 6—*ESEM micrographs of bacterial cells within biofilms.*

charging is dissipated into the gaseous environment of the specimen chamber, enabling direct observation of uncoated, nonconductive specimens. If water vapor is used as the specimen environment, wet samples can be observed directly, and EDS data can be collected at the same time as sample morphology and topography are photographed. Figure 7 shows a flow diagram for sample preparation for SEM compared to that for ESEM.

Corrosion and sulfide film formation on copper-containing metals can be followed using ESEM/EDS. In the presence of S^{2-}, a porous layer of cuprous sulfide with the general stoichiometry Cu_2-xS, $O < x < 1$ forms [*32*]. Copper ions migrate through the layer, react with more sulfide to produce a thick black scale. It has been argued that if the copper sulfide

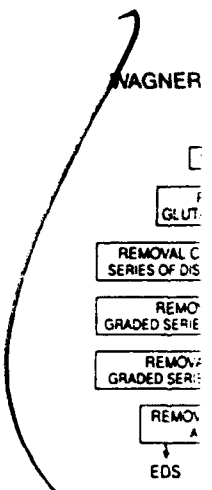

FIG. 7—*Flow ch*

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Dealloying of nickel from
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Several new forms of mi
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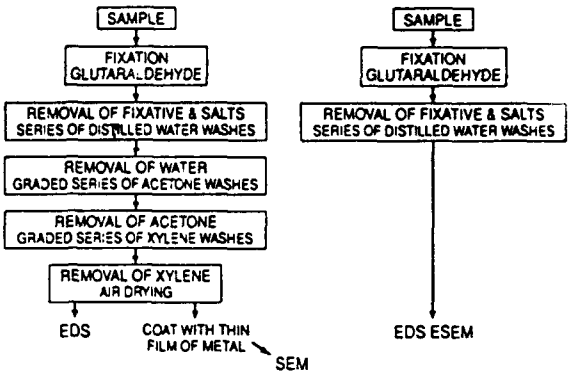

FIG. 7—*Flow chart comparing sample preparation for SEM and ESEM.*

layer were djurleite ($Cu_{1.96}S$) the sulfide layer would be protective. However, even if such a sulfide film were technically passivating, the film's mechanical stability is so poor that sulfide films are useless for corrosion protection. In the presence of turbulence, the loosely adherent sulfide film is removed, exposing a fresh copper surface to react with the sulfide ions. Preparation of microbiologically-produced sulfide corrosion products on copper foils for the SEM physically or chemically, or both, removes material from the surface.

An important feature of wet biofilms on copper-containing metals was that the microorganisms were distributed throughout the copper/nickel/iron-rich surface layers and not on top of these layers as some traditional scanning electron micrographs have indicated. Fixation, dehydration, and critical-point drying resulted in a loss of material from the surface so that many bacteria were removed with the surface deposits. It has been previously reported that bacterial cells attached to the base metal were tenaciously attached to the surface and were not removed or distorted during the SEM preparation [*31*]. TEM has been used to demonstrate that bacteria were intimately associated with the corrosion products and that on copper surfaces, the bacteria were found between layers of corrosion products and attached to base metal [*29*]. Similarly, ESEM images demonstrate that SRB were distributed throughout the sulfur-rich corrosion layers.

Dealloying of nickel from copper/nickel alloys and intergranular corrosion as a result of MIC has been reported by several investigators. Little et al. used EDS to demonstrate selective dealloying of Monel 400 in the presence of SRB from an estuarine environment (Fig. 8) [*30*]. The first evidence of a spatial relationship between the constituents of the biofilm and dealloying within pits covered with bacteria and diatoms has been presented. Little et al. demonstrated that diatoms are easily removed from marine biofilms during preparation for SEM and advanced the opinion that the role of diatoms in MIC has been neglected (Fig. 5) [*31*].

Several new forms of microscopy have been recently developed. Brief descriptions of those with potential application to the research study of biofilms follow.

Confocal laser scanning microscopy uses mechanical scanning of the object and a laser light source. A pinhole diaphragm just before the photomultiplier allows detection of light from very small specimen areas. High spatial resolution is achieved where horizontal optical sections can be collected and compiled for 3-dimensional image analysis [*33*]. Geesey has used this technique to study multidimensional images within a biofilm[2].

[2] G. G. Geesey, personal communication, 1992.

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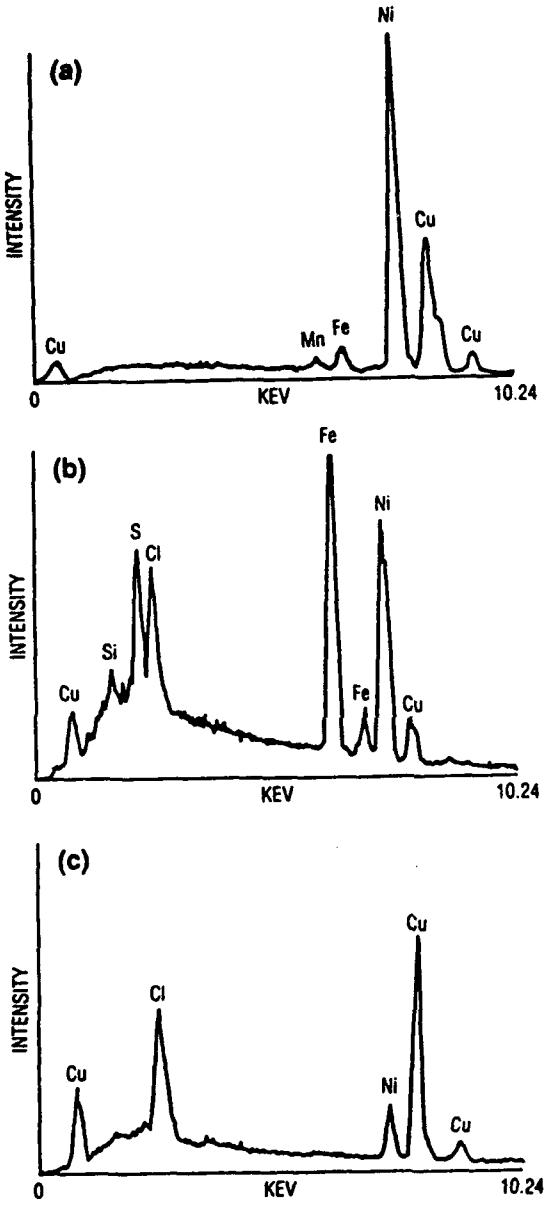

FIG. 8—*EDS spectra for nickel alloy Monel 400 before and after exposure to estuarine water* [*30*]: (*a*) = *unexposed*, (*b*) = *exposed for 6 months, showing accumulations of Si, S, and Cl with increased Fe and Ni, and* (*c*) = *residual metal in base of pit showing Ni depletion and Cu enrichment.*

Scanning tunneling microscopy uses the principle of quantum mechanical tunneling. The microscope tip and the sample form two electrodes between which tunneling can occur through a nonconductor, usually a vacuum, but can be other media such as water or an electrolyte. The tip moves in x,y,z dimensions to yield a surface map of local density states.

This technique has been app
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Chemistry

(1) Aqueous Medium

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(2) Base Metal and Corrosi

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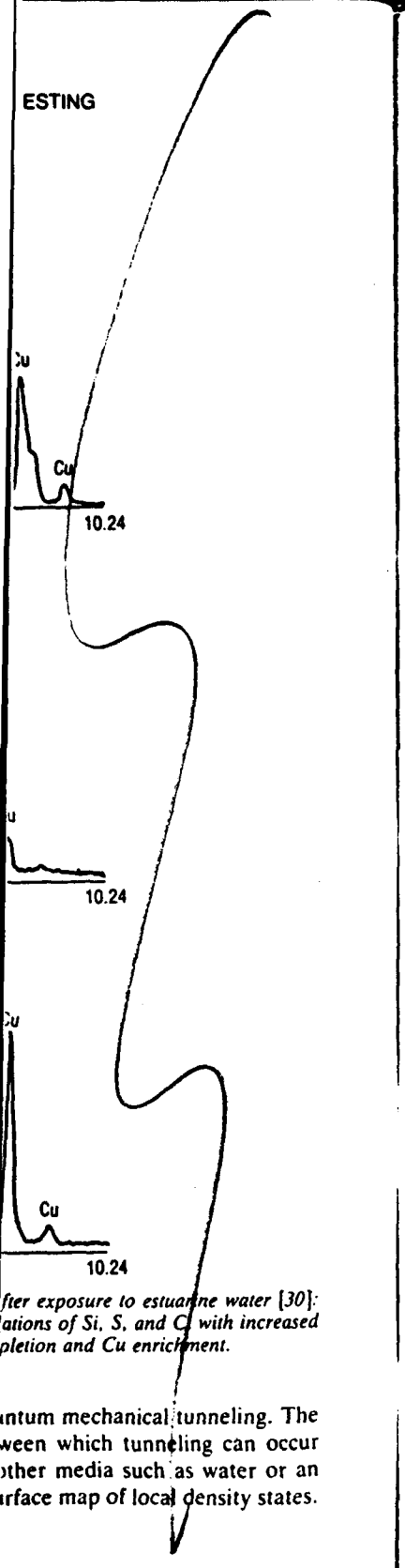

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This technique has been applied to imaging unstained, uncoated viruses [*34*]. Atomic force microscopy provides contour images, as used by Bremer et al., to show spatial relationship between a bacterium and a pit on a copper surface [*35*]. Probes use x,y,z piezotranslation to position a sample in contact with a microfabricated cantilever where it is scanned in a raster pattern.

Chemistry

(1) Aqueous Medium

Chemical analyses of the liquid medium from the corrosion system has been described previously and should be performed according to standard methodology.

(2) Base Metal and Corrosion Products

Elemental analysis of base metal and corrosion deposits using SEM/EDS or ESEM/EDS has been described. EDS spectra are obtained while the electron beam scans the area of interest to obtain true average compositions and avoid bias associated with selecting a specific point on an inhomogenous surface. Associated dot mapping shows distribution of an element of interest within a field. Figure 9 illustrates use of dot mapping in investigation of delam*ination of a zinc coating on galvanized steel*[3].

Scanning Auger microprobe analyses were used by Lee et al. to show metallic segregation in butt-welded 90/10 Cu/Ni piping [*36*]. The composition of the heat-affected zone was profiled by a series of overlapping images to allow calculation of percentage compositions from peak amplitudes. As an example, Fig. 10 shows a profile of percent concentration calculated from Auger data for carbon across the heat affected zone of a copper/nickel butt weld.

McNeil et al. analyzed sulfide mineral deposits on copper alloys colonized by SRB in an attempt to identify specific mineralogies that could be used to fingerprint SRB activity [*32*]. They concluded that the formation of nonadherent layers of chalcocite (Cu_2S) and the presence of hexagonal chalcocite were indicators of SRB-induced corrosion of copper. The compounds were not observed abiotically and their presence in near-surface environments could not be explained thermodynamically. Others have identified mackinawite, gregrite, and symthite as indicators for SRB corrosion of ferrous metals in anaerobic environments [*37*].

Sulfur isotope fractionation was demonstrated by Little et al. in sulfide corrosion deposits resulting from the activities of SRB within biofilms on copper surfaces [*38*]. ^{32}S was accumulated in sulfide-rich corrosion products and ^{34}S was concentrated in the residual sulfate in the culture medium. Conventionally, the amplitude of each isotope is not reported individually, but a ratio is established and compared to the isotope ratio of a standard to yield a value $\delta^{34}S$ expressed as parts per thousand. Negative δ values indicate a concentration of ^{32}S, and positive values indicate an accumulation of ^{34}S. Accumulation of the lighter isotope was related to surface derivatization or corrosion as measured by weight loss. Use of this and the preceding mineralogical technique to identify SRB-related corrosion requires sophisticated laboratory procedures.

[3] B. Little, unpublished work, 1987.

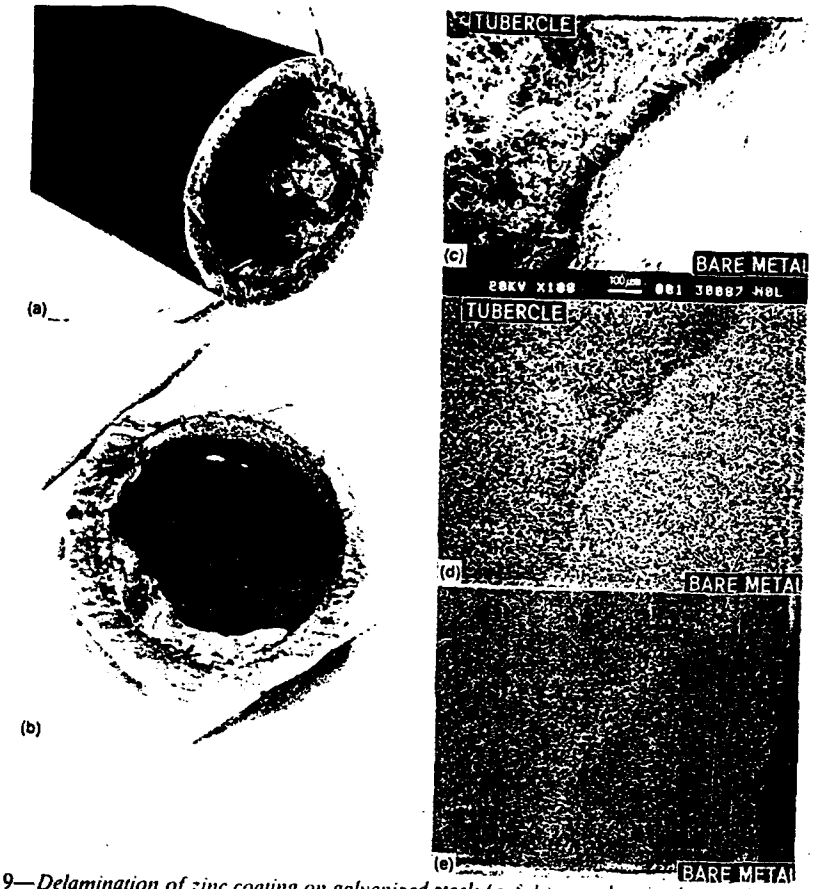

FIG. 9—*Delamination of zinc coating on galvanized steel: (a & b) = galvanized steel pipe containing tubercles of iron-oxidizing bacteria, (c) = SEM micrograph of tubercle, (d) = EDS dot map for Fe for area shown in (c), and (e) = EDS dot map for Zn for area shown in (c).*

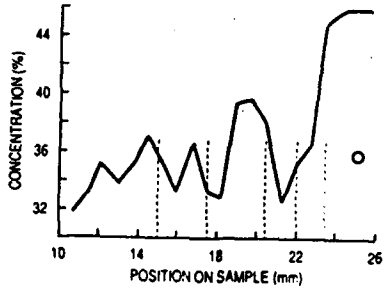

FIG. 10—*Profile of percent concentration of carbon across heat affected zone of a copper/nickel butt weld [36]. Weld bead starts at 22 mm and proceeds to the right, melt zone at approximately 16 mm.*

(3) Interfacial Chemistries

Including pH, dissolved
[9], and VanHoudt et al. [
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Metallurgy

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Conclusions

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Acknowledgment

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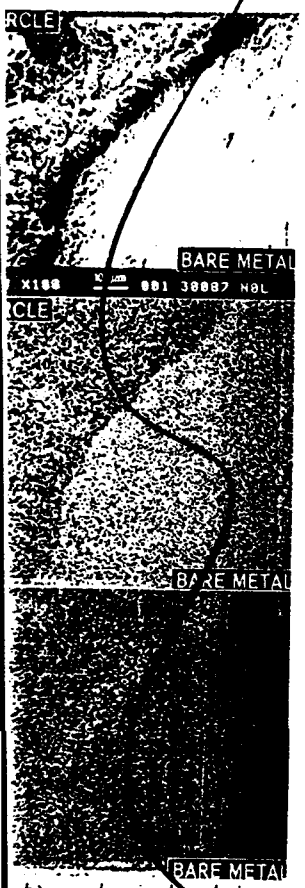

b) = galvanized steel pipe containing hercle. (d) = EDS dot map for Fe for on in (c).

affected zone of a copper/nickel butt elt zone at approximately 16 mm.

(3) Interfacial Chemistries

Including pH, dissolved oxygen, and sulfides have been measured by Lewandowski et al. [*9*], and VanHoudt et al. [*39*]. Microelectrodes were developed to profile these parameters systematically through the biofilm from external surface to the metal/biofilm interface.

Kearns et al. used X-ray photoelectron spectroscopy to characterize the interaction of metal ions and SRB for interfacial studies [*40*]. The biomass was irradiated to induce photoelectron ejection. By measuring kinetic energies of the photoelectrons, the binding energy of the electrons can be calculated. The binding energy value contributes to elemental identifications. Shifts in binding energy identifies oxidation state of the element.

Metallurgy

Alloy composition, mechanical properties, and microtopograph[illegible]e indicators of corrosion susceptibility. Examples of localized attack for specific all[illegible] been discussed earlier. Deformation of grain structure, presence of inclusions, and an[illegible]er manufacturing defects provide sites of decreased corrosion resistance [*6*]. In certain environments, heat-affected zones of welds in stainless steels are very susceptible to MIC [*10*]. In metallographic examination after exposure, identification of localized attack, carbides from graphitization, and hydrides from hydrogen-producing bacteria may suggest MIC.

Conclusions

In ideal circumstances, basic examination of a corrosion system includes preliminary knowledge of the corroding material and its operating environment. Initial visual observations of the undisturbed sample and subsequent microbiological, microscopic, chemical, and metallurgical examinations should provide reliable evidence for MIC.

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Jeffery R. Kearns and Brenda J. Little, Editors

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Foreword

The symposium on Microbiolo
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