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DOW

NUCLEAR SERVICES

TECHNICAL STUDY FOR
THE CHEMICAL CLEANING OF DRESDEN-1
DNS-D1-016

VOLUME II
Section 3

MASTER

THE DOW CHEMICAL COMPANY

MIDLAND, MICHIGAN 48640

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JUNE 15, 1977

Prepared by:

DOW NUCLEAR SERVICES

DOW CHEMICAL COMPANY U.S.A.

MIDLAND, MICHIGAN

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CORROSION STUDIES - FINAL REPORT
on
DOW INDUSTRIAL SERVICES - DOW SOLVENT
NS-1 CLEANING of COMMONWEALTH
EDISON COMPANY'S DRESDEN NUCLEAR POWER
STATION - 1

July, 1975

by

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ABSTRACT

Alloys representative of those used to fabricate the boiling water reactor (BWR) of the Dresden-1 Nuclear Power Station were subjected to corrosion trials in the proprietary solvent, NS-1, developed at the Dow Chemical Company. The trial conditions usually were on a "worst possible case" basis with respect to time, temperature, degree of alloy sensitization, and level of stress compared to the conditions which actually would exist during the decontamination of Dresden-1 with the solvent.

These corrosion trials included specific tests for general corrosion, stress corrosion cracking, intergranular corrosion, and crevice corrosion, as well as tests of galvanic couples. The results led to the conclusion that serious corrosion should not occur as a result of decontaminating the BWR components with Dow solvent NS-1 for 100 hours at 250° under nitrogen padding.

Because Dresden-1 contains sensitized 304 stainless steel, albeit in small localized areas, a large part of the NS-1 corrosion study was devoted to determining if the solvent would cause intergranular stress corrosion cracking (IGSCC) of severely sensitized and stressed 304 stainless steel.

Tests of 304 stainless steel specimens of many different configurations and stress levels under various conditions led to the conclusions that:

1. Dow solvent NS-1 will not produce IGSCC in Dresden-1, and
2. Dow solvent NS-1 is not more aggressive than deionized water under the same conditions.

Type 304 stainless steel will exhibit intergranular stress cracking in Dow solvent NS-1, however, if:

1. the alloy is sensitized for >30 hours at 1200°F and is not descaled before it is exposed to either Dow solvent NS-1 or deionized water.
2. the alloy is sensitized for >30 hours at 1200°F in an inert atmosphere, descaled, and exposed to either Dow solvent NS-1 or deionized water which contains >0.12 wt% Fe⁺³ or ~0.1 wt% Cr⁺⁶.

The above conditions caused stress cracking in U-bends of 304 stainless steel when the specimens were bent after they were sensitized. When the U-bends were formed before the alloy was sensitized, with one or both of the above conditions present, no stress cracking occurred. No other specimens of sensitized 304 stainless steel U-bends exhibited intergranular cracking in Dow solvent NS-1.

No excessive general corrosion rates, crevice corrosion, galvanic corrosion coupling effects, or other localized attack occurred when the alloys characteristic of those found at Dresden-1 were exposed to Dow solvent NS-1. The tests indicated general corrosion rates of <5 mpy can be expected on all 300 series stainless steels. These rates would yield a maximum metal loss of 0.06 mils at the end of the decontamination. The tests also indicated maximum total metal losses of 1-5 mpy can be expected on 1020 carbon steel, low alloy steels, 2-1/4 Cr - 1 Mo alloy, and 400 series stainless steels as a results of the decontamination; losses of <0.1 mils are anticipated on copper, nickel, and chromium based alloys and on the specialty alloys.

Decomposition of NS-1 solvent trapped in artificial crevices of sensitized and stressed 304 stainless steel under hydro-test conditions (500°F in deoxygenated, deionized water) did not produce unacceptable attack on the alloy. Analyses indicated thermal decomposition of Dow solvent NS-1 did not produce potentially harmful breakdown products.

When valve packing, FLEXITALLIC^a and asbestos gasket materials were exposed to Dow solvent NS-1 in conjunction with sensitized and stressed 304 stainless steel, no materials harmful to the 304 alloy were leached from the nonmetallic components. GRAPHITAR^b 14, a nonmetallic material used for bearings in the main coolant pumps at Dresden-1, was inert when exposed to NS-1 solvent under dynamic test loop conditions.

3.0 INTRODUCTION

Extensive trials were conducted by the Contract Research Department of the Texas Division of the Dow Chemical Company for Dow Industrial Service (DIS) to evaluate the potential corrosion effects from

^aTrademark of Flexitallic Gasket Company

^bTrademark of Wickes Engineered Materials Division

decontamination of Commonwealth Edison Company's (CECO's) Dresden-1 Nuclear Power Station with a proprietary cleaning solution, Dow solvent NS-1.

Decontamination is required in order to reduce radioactivity levels so that improved inspection and maintenance can be performed on this nuclear facility which has been in service since 1960.

The corrosion research program was carried out from September, 1973, to April, 1975. The program covered several thousand individual corrosion tests of all the basic Dresden-1 BWR construction materials as they were exposed to Dow solvent NS-1 under conditions of time and temperature exceeding those proposed for the actual decontamination. Over 8,000 technical employee-hours were spent in evaluating general weight loss, corrosion by examination of pitting, crevice, and galvanic corrosion effects and stress corrosion cracking for each major metal or alloy family in the CECO system.

Several isolated cases of intergranular stress corrosion cracking (IGSCC) of stressed and sensitized type 304 stainless steel have been reported in the literature, including those which occurred at Dresden-1. The small number of failures reported were attributed to areas in the reactor which had a high degree of sensitization and cold work (welds). Therefore a significant amount of this study was concerned with measuring the potential IGSCC effects of Dow NS-1 solvent on sensitized and stressed 304 stainless steel.

The majority of the research on this solvent was performed by Dow Chemical personnel. However, two other research groups conducted independent evaluations of Dow solvent NS-1.

Professor Roger Staehle, of the Ohio State University, an independent consultant for Commonwealth Edison, carried out electrochemical and slow strain rate tests of Dow solvent NS-1 (Appendix VI). His results substantiate the major findings of this report, and were formally reported to Commonwealth Edison.

The Nuclear Energy Division of General Electric, San Jose, California, has performed independent stress corrosion tests on highly sensitized 304 stainless steel exposed to Dow solvent NS-1 according to the procedure listed in Appendix V.

The independent tests by Professor Staehle and the General Electric personnel contributed greatly to the

corrosion studies reported in this document. Dr. Craig Cheng of the Argonne National Laboratory gave significant advice for the preparation of the corrosion test matrix (see Appendix II of this report).

This report summarizes the general approach to the corrosion studies, the experimental procedures used, and the corrosion trial results. Detailed accounts are recorded in data books of the Dow Chemical Company, Texas Division, and are on permanent file at Freeport, Texas. All exposed metal samples will be retained for a reasonable length of time after successful completion of the decontamination of Dresden-1. The trial results for each type of corrosion on each major alloy and/or alloy class as well as metallographs and discussion of the results are presented in this report. Individual "Corrosion Trial Data Sheets" for some of the more significant tests are included in Appendix I to provide additional technical information.

3.1 APPROACH

3.1.1 Materials Review

DIS personnel in conjunction with CECo and Dow Contract Research representatives made a comprehensive study of all drawings and specifications for Dresden-1. All materials in the system were listed by the American Society of Mechanical Engineers (ASME), American Society of Testing Materials (ASTM), or other specification numbers and by areas of use. From this listing, various alloys representative of all classes of materials in the system were chosen for corrosion testing. The testing of these alloys was weighted to reflect the amount of alloy present in Dresden-1 and the importance of the alloy based on its location in the structural components of the system. A summary of the types of major construction materials used at Dresden-1 is given in Section 3.10.1.

3.1.2 CORROSION TRIAL MATRIX

A corrosion trial matrix was developed as a result of joint meetings with DIS, CECo, and CECo consultants, Dr. Roger Staehle of Ohio State University and Dr. Craig Cheng of Argonne National Laboratory. This matrix related every material to be tested with: (1) the form of the material (wrought, welded, cast, forged, etc.); (2) type of heat treatment and condition of the surface (annealed, sensitized, hardened, precipitation hardened, scaled, descaled before exposure, etc.); (3) type of corrosion trial (general

corrosion, effect of galvanic couples, crevice, pitting, stress corrosion cracking, etc.); and (4) environmental modifications to be considered in each trial aeration; nitrogen padding; deliberate contamination with heat-produced scale, metal and ions such as Cl^- , F^- , Pb^{+2} , or S^{-2} ; use of reducing or oxidizing environment, or static or dynamic conditions etc.).

3.1.3 CONTINUING CORROSION TRIAL PLAN DEVELOPMENT

Frequent intercommunication among DIS, CECO, and CECO consultants provided continuous updating of corrosion trials. Occasionally tests of an alloy provided enough data early in the study so that further testing of the alloy was not warranted. More often the updating resulted in increasingly sophisticated corrosion trial methods or indicated that additional environmental or metallurgical considerations were required.

3.1.4 WORST POSSIBLE CASE BASIS

The decision was made at the start of the corrosion study to perform testing on a "worst possible case basis." Both advantages and hazards exist in this approach: namely, metals and alloys which pass such testing will not fail under "most probable operating case" conditions, and conversely, failures no matter how few, which occur during the testing, even under highly exaggerated conditions, tend to outweigh further results. Subsequent tests which are only slightly less severe and produce no failure do not erase the doubt of possible "in-service failure."

In this corrosion study, "worst possible case conditions" included:

- Sensitization of alloys at temperatures 100-150°F above those used in actual construction of the reactor facility.
- Sensitization of alloys for times of up to 50 hours.
- Stressing the alloys after they were sensitized, with the brittle carbide precipitates surrounding the grain boundaries in place, as opposed to actual practice where components were stress relieved after they were fabricated and stressed.

- Stressing the alloys to levels grossly in excess of their yield points.
- Exposure of specimens to NS-1 solvent at 25°F higher than expected maximum operating temperatures.
- Exposure of specimens for 25-300% greater than expected cleaning times.
- Exposure of heavily scaled specimens whereas Dresden-1 components actually were cleaned and free of heat-treatment scale before the unit was put into operation.
- Exposure of specimens to NS-1 solvent which contained contaminants at much higher concentrations than would be present during the actual cleaning operation.
- Use of configurations such as those found in double U-bend stressed specimens and severely creviced specimens, which would not be found in an operating unit and that are particularly prone to produce adverse corrosion effects.

Testing on this worst possible case basis demonstrated that the majority of the alloys at Dresden-1 will resist significant corrosion by Dow solvent NS-1. In those few cases where specimens showed some susceptibility to corrosion, the specimens were retested under conditions which approached a worst possible case basis but were closer to the most probable operating case basis. These worst possible case conditions were used during the initial phase of corrosion screening. Alloys that successfully passed such extreme corrosion tests were eliminated from the test matrix so that more time could be spent evaluating alloys which appeared susceptible to corrosion.

Negative results are difficult to prove in corrosion testing. Unanswered questions remain even after tests of long duration: e.g., would the specimen have failed if the test had been extended for a longer period of time, or did the test environment inhibit the corrosion mechanism under study? Answering these questions

directly by experimentation is time consuming; therefore the approach taken in this study involved testing the alloys under conditions known to cause failure. Once the results from these test conditions were well understood, the severity of the conditions was relaxed by a measured amount and the test was repeated. Subsequent results then were used to judge the probability that a particular corrosion mechanism would operate during the actual decontamination of Dresden-1.

For example, some 304 stainless steel specimens which were oversensitized and overstressed in relation to the alloys found in Dresden-1 underwent intergranular stress corrosion cracking in either Dow solvent NS-1 or deionized water under sufficiently severe conditions of heat treatment, stress levels, contamination levels, and configuration. However, specimens tested under identical conditions, but with the amount of cold-worked areas reduced, showed no tendency toward such IGSCC, even when stressed to well beyond their yield points or when highly sensitized by exposure at temperatures and time periods in excess of those anticipated for the actual cleaning.

3.2 EXPERIMENTAL PROCEDURES

Corrosion testing by its nature precludes complete standardization. The following are descriptions of test methods which were adapted as a guide so that some of the pitfalls of such testing would be avoided. The tests described herein probably are the best means available for evaluating the behavior of metals in service, short of actually constructing and operating a piece of equipment.

In all corrosion testing the volume of solvent to surface area of metal exposed was maintained at a ratio of one (1) gallon to one (1) square foot, approximately that found in the Dresden-1 reactor.

The majority of testing was performed under static conditions in an effort to enhance localized corrosion mechanisms. Since the actual cleaning will be of short duration the most important corrosion mechanisms are those which act in the shortest time: i.e., pitting, crevice, and stress corrosion.

Preliminary tests indicated that specimen surface roughness was not a highly significant factor in the corrosion rates obtained with Dow solvent NS-1. Therefore the specimens which had fairly smooth surfaces, were tested as received from the supplier, and were not subjected to surface polishing.

The shape of the coupons chosen for most of the corrosion tests and the volume of solvent necessary to yield the correct coupon surface to volume ratio dictated that a tall narrow test container be used. Two types of containers were selected. A 9" x 2" PYREX^a glass tube was used as the coupon container when oxygen concentration was a test parameter. The tubes were sealed with blind glass flanges joined via metal clamps with a TEFLON^b gasket between the faces of the flanges. Glass valves and a gas sparger were incorporated into the top of the glass tubing. A 9" x 2" TEFLON lined pipe flanged on each end was used as the test container when air or oxygen concentration was not a test parameter (Figure 3.2). By using the flange designs noted, the test samples are isolated such that they cannot be affected galvanically by the test containers.

The compatibility of the test containers with Dow solvent NS-1, was evaluated to determine if fluoride ion was leached from TEFLON by the solvent, thereby creating a potentially serious stress corrosion environment for the stainless alloys under test. Likewise, tests were made to determine if SiO₂ leached from the glass of the test container into the solvent since silicon dioxide inhibits corrosion of some alloys and could cause deceptively low corrosion rates to occur in alloy tests. All leach tests gave negative results on both glass and TEFLON (see Section 3.3.1.2).

The temperature of the test media was controlled by placing the test containers in a forced air convection oven capable of maintaining the temperature with a range of at least $\pm 5^{\circ}\text{F}$. For safety, a temperature sensor was installed on each oven to prevent excessive temperatures and subsequent pressure buildup in the test containers if the primary temperature control failed.

^aTrademark of Corning Glass Works

^bTrademark of the DuPont Company

Figure 3.2 Test containers used in weight
loss corrosion rate determinations

3.2.1 General Corrosion and Weight Loss Specimens

Most of the general corrosion weight loss data reported in this study were determined for specimens measuring 6" x 0.75" x 0.063". The thicknesses of some stainless alloys varied from 0.063-0.125" depending on the particular heat from which the specimens were obtained. Since the edges of sheared metal specimens occasionally undergo considerable attack by solvents, the edges of coupons used in this study were sheared in order to provide worst possible case conditions.

The use of duplicate specimens ordinarily is sufficient to give an accuracy of $\pm 10\%$ in laboratory corrosion tests under standardized conditions. Each weight loss datum point in this report, however, represents three to five specimens and thus provides an improved degree of confidence.

Weight loss specimens were stamped with identification and were treated as follows:

1. Mild steels and low alloy steels

Mild steel and low alloy steel specimens were precleaned by degreasing them in acetone. The alloys then were etched in 50% hydrochloric acid at ambient temperature to give reproducible initial surfaces as similar as possible to actual reactor surfaces. These pickled specimens were rinsed with distilled water and were dried with an acetone - air rinse. Each specimen was weighed and its surface dimensions were measured. The specimens then were exposed to the appropriate test media.

Exposed specimens were removed from the test solution, scrubbed with fine pumice under running water, rinsed in distilled water, and dried with an acetone rinse.

2. Stainless steel alloys

Since surface conditions on stainless steels can affect both their mechanical properties and corrosion resistance, stainless alloys with uniform surface finishes were obtained for testing. Each stainless specimen had a 2B surface finish which is produced by cold-rolling the steel to a specified thickness on either a handsheet mill or a continuous mill. Specimens subsequently are annealed, descaled, and given a light, cold-rolled pass on polished rolls.

Stainless alloys were pretreated by scrubbing them with a soft-bristled brush and fine pumice under running water in order to give a reproducible surface finish to the alloys. The

specimens then were weighed and exposed to the appropriate test media, scrubbed as above, dried with an acetone rinse, and reweighed.

3. Copper alloys

Copper alloys initially were pickled in 10% nitric acid at ambient temperature for one minute to give a reproducibly active surface. Each specimen was rinsed with distilled water, dried with acetone, weighed, and then exposed to the test media. The exposed specimens were cleaned with a soft-bristled brush under running water, rinsed with acetone, dried in an oven, and weighed.

Corrosion rates of the specimens were calculated from the following formula:

$$CR = \frac{(1.44/\rho)(\text{wt loss})}{(\text{area})(\text{length of test})}$$

where CR is the corrosion rate in mils/year (mpy), ρ is the density in gm/cm³, the weight loss is in milligrams, the area is in dm², and the length of the test is in days.

After its final weighing, each specimen was carefully examined for type and uniformity of surface attack. The number, size, and distribution of pits as well as their general shape and uniformity of occurrence were noted. The maximum and minimum depths of pits were measured with a calibrated microscope or depth gauge. A distinction was made between pits which occurred under spacers and on more boldly exposed surfaces.

3.2.2 Galvanic Corrosion Specimens

Galvanic effects are due to the flow of current between metals of dissimilar potential which are connected electronically and electrolytically. Certain variables must be held constant in order to obtain reproducible results in a galvanic test. The anode to cathode area ratio and the geometrical arrangement (distance apart of the specimens) are two important parameters that must be controlled before accurate galvanic corrosion results can be obtained. The galvanic effect is accentuated when the cathode is large in relation to the anode. Therefore, the relative areas in galvanic couples should be similar to those which occur in actual service. A 1:1 ratio was used in this study when the actual area relationship was unknown.

Three geometrical arrangements were chosen to approximate the different types of galvanic couples found in service. Some specimens were held together with rubber bands to produce both a crevice and a galvanic relationship. Insertion of a nonconductive washer from the specimens. The electrolytic resistance of the test solution will significantly affect the flow of galvanic current between the specimens in both of these types of couples. The third configuration consisted of dissimilar metals butt welded together which allowed galvanic effects on a weld zone to be studied. These types of couples were expected to represent any in-service galvanic couple more than adequately.

No extended galvanic tests were made since the actual cleaning of Dresden-1 is expected to require only 100 hours. Short-term galvanic tests can be misleading, however, in the presence of film effects, such as passivation or protection by an inhibitor, which proceed at different rates on different specimens. Additionally, the initial polarity of specimens may reverse after they undergo prolonged exposure to a test medium so that laboratory assessment of corrosion can be used only to predict whether galvanic corrosion in the field will be severe, moderate, or slight.

3.2.2.1 Welded Specimens

Galvanic coupons were made by joining two 3" x 3/4" x 1/16" pieces of dissimilar metal with 308 stainless steel weld rod. The weld bead was removed by grinding it flush with the specimen surface which was then sandblasted with 80-120 mesh polishing silica to give a uniform surface. Each sandblasted specimen was scrubbed with pumice under running water, dried with acetone, and weighed. All weight losses were assumed to be on the anodic member of the couple.

3.2.2.2 Bolted Specimens

Bolted specimens were made in a configuration which simulated that of a galvanic couple formed through mechanical contact of two dissimilar metals. The metals of the couple were separated by a slotted TEFLON spacer to prevent crevice effects. A bolt on each end of the specimen provided electrical contact. These specimens were cleaned in an acid solution, scrubbed with pumice under running water, dried with acetone, and weighed. Weight losses on both the anodic and cathodic member of the couples were measured.

3.2.2.3 Rubber-banded Specimens

A bimetallic galvanic couple can be formed easily by joining two specimens together with rubber bands. The anode to cathode area ratio is varied by cutting the specimens to different sizes and by placing the surface of one metal next to that of another metal to give a bimetallic crevice. The corrosion rates obtained from these specimens are not completely galvanic in origin but are derived from crevice effects. The corrosion products from one metal can, of course, accelerate attack on the other metal.

A welded bimetallic junction which contains a crevice is a potentially important type of specimen for this study. The structural support in the Dresden-1 pressure vessel consists of a low carbon steel alloy overlaid by stainless steel for protection against corrosion. If cracks exist in this cladding, a bimetallic crevice forms which has a large cathodic area (304 steel overlay) in contact with a small anodic area (pressure vessel steel).

A special specimen was designed to approximate this potentially hazardous condition. A 12" x 6" 304 stainless steel plate was used to simulate the cladding material. A small piece of low alloy steel was welded onto the center of the large plate to give an area ratio of 20:1. The specimen was welded on only three sides, leaving a crevice opening along the fourth edge. This specimen design should allow a conservative estimate of the galvanic action to be expected from exposing a crack in the cladding to the cleaning solvent.

Coupons exposed to the test media were cleaned, reweighed, and visually inspected as set forth in Section 3.2.1.

3.2.3 Crevice Corrosion Specimens

Crevice corrosion occurs in two separate steps: initiation of attack followed by propagation of the attack. Separate specimen designs are necessary to study both steps as they relate to the cleaning solvent. The artificial crevice produced by the TEFLON spacer bolted to the surface of a metal plate or strip allows the susceptibility of a metal to initiation of corrosion in a crevice to be determined. The TEFLON is inert and yet restricts diffusion of the solvent so that crevice attack can occur if the specimen is inherently susceptible to this type of corrosion.

The propagation phase is less easily studied and quantified than is the initiation phase. Two important questions about crevice propagation must be answered by laboratory testing: (1) how will the cleaning solvent interact with an already existing crevice; and (2) will the thermal decomposition products from the residual cleaning solvent, which will decompose at less than plant operating temperatures, degrade the existing crevice after the equipment returns to service? More than one specimen configuration was employed to answer these important questions.

3.2.3.1 TEFLON Artificial Crevice Specimens

Two basic types of TEFLON-produced crevices were employed to study initiation of crevice attack. One type consisted of a TEFLON shoulder washer bolted on to the surface of a metal strip. If initiation of crevice attack occurred, a roughened area appeared beneath the shoulder washer. The depth of attack was measured with a calibrated microscope by focusing first on the uncorroded metal near the crevice and then on the bottom of the roughened, corroded area of the crevice. Accurate depths of crevice attack were measured with an optical micrometer.

The other type of artificial crevice specimen was a variation of the shoulder washer specimen. Thirty-two small crevices were machined into a TEFLON base. The test metal was cut from sheet or plate into 3" x 3" squares and the sheared edges were ground to eliminate crevices inherent in rough, machined or sheared edges. The machined TEFLON was attached to the center of the 3" x 3" plate and was bolted down to a uniform torque of 20 foot-pounds, after which the specimens were exposed to the test media for up to 300 hours (Figure 3.2.3.1).

Figure 3.2.3.1 - Teflon multi-crevice assembly with stainless specimens.

Measurements were made to evaluate (1) the percent of crevices in which initiation began, (2) the mean depth of attack for crevices in which corrosion occurred, (3) the maximum depth of attack, and (4) the average weight loss. From the data, various statistical probabilities can be developed, i.e., the probability of attack developing in a specified time for a structure having a limited number of crevices, the probability of not exceeding a specified depth of attack during a given exposure, etc.

3.2.2.2 Rubber-banded Specimens

These crevice specimens were similar to those described under galvanic corrosion, Section 3.2.2.3, except that both parts of each specimen here had the same composition.

Two flat metal coupons were held together with rubber bands. A small milled area was machined on one coupon to give a crevice where solvent could be sequestered. This type of crevice specimen was heat treated and cycled for 100 hours at 250°F (the conditions planned for the actual decontamination). After it was cycled, the specimen was transferred to an autoclave that contained deoxygenated, distilled water.

The transfer was made so as to ensure that cleaning solvent remained trapped in the milled portion of the specimen. The autoclave was operated at 500°F for 100 hours to ensure thermal decomposition of the trapped solvent, after which the weight loss of the specimen was measured.

3.2.3.3 Fitted Tubing specimens

Larrick and Weed studied cracking which resulted from entrapment of alkaline permanganate in stressed crevices by testing a simple crevice specimen as well as a more complex fitted tube of the type shown in Figure 3.2.3.3.1. The simple crevices consisted of two flat strips of metal joined together; one strip in each pair previously had been machined to contain a 5 mil-deep slot. The crevice specimens were stressed with a deflection beam.

Larrick and Weed found that the type of crevice in which the solvent was trapped affected whether or not caustic-produced cracking occurred. The specimens with simple crevices did not exhibit cracking in alkaline permanganate solutions over a five year test period, whereas the complex tubing specimens underwent failure by stress cracking after only two to four weeks of exposure.

Figure 3.2.3.3.1 - Typical fitted tubing assembly used for crevice evaluation of Dow Solvent NS-1.

Simple crevices allow diffusion and mixing of the solution so that conditions conducive to cracking may not be achieved. The fitted tubing type of specimen was used in the present study in order to evaluate the corrosive effects of Dow solvent NS-1 under complex crevice conditions which might exist in Dresden-1.

Sensitized (1200°F, 50 hours, in air) 304 stainless steel SWAGELOK^a fittings and tubing were assembled while they were submerged in Dow solvent NS-1 so that approximately 0.3-0.5 ml of solvent was trapped in the two crevice areas of the assembled specimen. One crevice was designed so that it could be closed to completely trap the solvent; the other crevice allowed diffusion of the solvent. These specimens were exposed to Dow solvent NS-1 for up to 300 hours at 250°F under static conditions, after which they were placed in an autoclave that contained neutral distilled deoxygenated water for 100 hours at 500°F (Figure 3.2.3.3.2).

3.2.3.4 Electrochemical Initiation of Crevice Attack

Several electrochemical techniques have been used to investigate the localized attack that occurs on some corrosion resistant stainless steel and aluminum alloys. To date, none of the published techniques provide a foolproof quantitative method for predicting the extent to which an alloy will pit or crevice corrode in a given environment. A number of investigations based on polarization scanning techniques do provide useful information regarding crevice initiation. This method originates from observations that under conditions of cyclic potentiodynamic anode polarization, stainless steels exhibit active pitting above a specific potential defined as the critical pitting potential, E_{cpp} . Measuring this breakdown potential and comparing E_{cpp} to the open circuit potential (E_{ocp}) of an alloy gives a qualitative measure of the pitting tendency of the alloy. The lower the E_{ocp} of a metal, compared to its E_{cpp} , the less likely pitting will occur.

Figure 3.2.3.4.1 represents a typical polarization curve obtained for 304 stainless steel (SS) in water as an example. The specimen reached its open circuit potential and then exhibited a sudden increase in anodic current. The breakdown potential is determined from this polarization curve. The forward anodic polarization curve is extrapolated to E_{ocp} ; the general corrosion rate then can be calculated from the corrosion current, i_c , by use of Faraday's Law.

^aTrademark of Crawford Fitting Company

Figure 3.2.3.3.2-High temperature autoclave used for testing the corrosive nature of the decomposition products of Dow Solvent NS-1.

Area A - Pitting Attack Initiates
and Propagates
Area B - Pitting will not Initiate
but will Propagate
Area C - Pitting will not Initiate
or Propagate

E_{cpp} - Critical Pitting Potential

E_{pp} - Passivation Potential

E_{ocp} - Open Circuit Potential

Figure 3.2.3.4.1 - Schematic representation of a typical polarization curve of 304 SS in sea water

This method was adapted to the evaluation of crevice attack. An artificial crevice was produced by placing a rubber O-ring snugly around a test electrode to produce a region of locally limited diffusion and thus a weakened area for breakdown of passivity. (Figure 3.2.3.4.2). A leakproof assembly was obtained by a proper compression fit between the electrode and a TEFLON gasket. The working electrode, test electrode and reference electrode were mounted in a one liter flask as shown in Figure 3.2.3.4.3. The working electrode consisted of one square centimeter of platinum; the reference was a saturated calomel electrode. An M-4100 PETROLITE^a potentiodyne analyzer was used to control the potential of the test electrode. The test electrode was allowed to equilibrate (usually 18 hours) and was anodically polarized from its rest potential at a rate of 10 millivolts per minute.

The polarization scan for each specimen was measured up to at least 700 millivolts above the rest potential. No attempt was made to exclude air from the test cell; the test was carried out at 200°F.

The electrode was visually examined for evidence of crevice initiation beneath the O-ring after each test.

3.2.4 Intergranular Stress Corrosion Cracking Specimens

Stress-induced corrosion is one of the most insidious forms of corrosion. The conditions associated with cracking are specific to a given alloy system. However, certain general features have been observed, four of which are listed here:

1. Alloys subject to cracking normally are considered passive and noncorroding in many environments, and corrode uniformly at rates of less than five mils per year.
2. Species which cause stress corrosion often are those which appear innocuous in identity or concentration.
3. Stress-induced corrosion can propagate with no externally applied stress from stresses produced when specimens are welded, formed, or machined.
4. The conditions leading to stress corrosion are specific to a given alloy.

^aTrademark of Petrolite Corporation

Figure 3.2.3.4.2-Electrode holder for crevice initiation evaluation.

Figure 3.2.3.4.3- Electrochemical test equipment for cyclic polarization.

The last feature allows engineering use of alloys susceptible to stress corrosion. Enveloping the environment responsible for stress corrosion allows the alloy to be successfully employed outside the envelope. Creating this envelope requires performing properly planned corrosion tests in the anticipated environment.

Specimen designs for testing stress corrosion vary from laboratory to laboratory, but generally can be divided into two groups, depending on the method used to stress the specimens. In one method, a constant load is applied to produce tensile stresses over the cross section in an axial direction or in the outer fibers of a bent specimen. In the second method, stresses are induced by subjecting the specimen to a predetermined constant deflection. Both types of specimens were represented in the evaluation of the stress corrosion characteristics of alloys exposed to Dow solvent NS-1.

3.2.4.1 Single and Double U-Bends

U-Bend specimens contain large amounts of both elastic and plastic strain. Alloys fabricated in this configuration are subjected to some of the most severe conditions used for testing smooth (as opposed to notched or precracked) stress corrosion specimens. When U-bends are fabricated, an undefined amount of stress is imposed on the specimens.

The stress of principal interest is circumferential, and is nonuniform because:

1. a stress gradient through the thickness of the specimen varies from maximum tension on the outer surface to a maximum compression on the inner surface.
2. the stress varies from zero at the ends of the specimen to a maximum at the center of the bend, and
3. the stress varies across the width of the specimen.

The total strain on the outside of the specimen is approximated closely by:

$$\xi = T/2R \text{ when } T \ll R$$

where ξ is the total strain,

T is the specimen thickness, and

R is the radius of bend curvature.

The dimensions of the specimens, especially the ratio of thickness to bend radius, should be kept constant so that approximately the same maximum strain will exist in the alloys being compared. Specimens are cut from sheets or plates either transversely or longitudinally to the direction in which the original piece was rolled. Often the stress corrosion resistances of the resulting specimens are quite different, requiring that the orientation of the specimens be defined for test purposes.

Most of the U-bend specimens tested in this study were fabricated in two stages from rectangular strips $6'' \times \frac{3}{4}'' \times \frac{1}{16}''$ to $\frac{1}{8}''$ in size. However, some specimens were fabricated from 2'' wide strips rather than $\frac{3}{4}''$ wide. The strips were bent with a $\frac{3}{4}''$ radius into an approximate U-shape, after which the elastic strain was allowed to relax completely. The test stress was applied in the second stage of fabrication. TEFLON shoulder washers were used in bolting the ends of the specimen together to prevent galvanic coupling. In this way the specimens were held under constant strain during the stress corrosion tests.

The surfaces of the U-bends contained a 2B mill finish, which is a smooth, bright, cold-rolled finish.

The U-bend apparatus and a typical U-bend specimen are shown in Figures 3.2.4.1.1 and 3.2.4.1.2, respectively.

Stressed specimens were degreased and examined for mechanical cracking prior to being tested. Specimens usually were heat treated before they were bent, although some were heat treated following the first stage of fabrication. Heat scale was removed from some specimens by pickling them in Dow solvent NS-1 exposed to air at reflux temperatures. When possible, coupons were pickled before they were bent, so that the final stress was not applied to scaled specimens.

Double U-bends were fabricated in a manner similar to that used to form single U-bends except that two rectangular coupons were bent simultaneously instead of one. Fabrication of double instead of single U-bends gave more severely stressed specimens because of the resulting crevice which existed at the interface of each coupon. Galvanic coupling can be imposed on a double U-bend by fabricating it from coupons of dissimilar alloys to give a specimen which is simultaneously subjected to stress, crevice, and galvanic effects. Such a specimen may be used to simulate actual in-service conditions.

Figure 3.2.4.1.1 - Assembly for U-bending different types of specimens.

Figure 3.2.4.1.2 - Single U bend specimen
fabricated in 2 stages.

After the specimens were exposed to the test media, they were examined visually and were released from the test stress. Each specimen was examined under low magnification with a stereo microscope. In tests where no cracks occurred, specimens were examined metallographically at 100-500X magnification.

3.2.4.2 Jig-Loaded Tensile Rods and Dog-Bone Specimens

Specimens for constant strain corrosion tests were fabricated from 4-1/2" lengths of 304 stainless steel tensile bar stock that had been machined from a diameter of 3/8" to a constant cross-section of 1/4". The end of each rod was threaded to fit a stressing jig which was made of 304 stainless steel to prevent galvanic coupling effects from occurring in the specimens. "Dog-bone" shaped specimens were fabricated from low alloy type SA 516 Grade 70 steel and 405 stainless steel since these metals were obtainable only in sheet form. An adapter was made to fit these specimens into the stressing jig. The jig and specimens are shown in Figure 3.2.4.2.1.

Test results with these specimens were used to supplement those obtained with U-bend specimens. However, in contrast to the U-bend specimens, tensile rod and dog-bone specimens contained a defined rather than an unknown amount of cold-work and stress. The constant total strain specimens were loaded initially into a conventional tension tester and were strained to the desired level. A restraining jig made of the same alloy as the specimen was then placed around the specimen and was tightened until the load was carried entirely by the jig. An assumption was made that the strain in the specimen was retained as the specimen was transferred from the tension tester to the jig since the jig frame was massive compared to the loaded specimen. The surface area of 304 stainless steel in the jig supporting the load was at least 13X greater than the cross-sectional area of the specimen resisting the load.

The way in which the surface on a constant strain specimen is prepared can markedly influence the results obtained in stress corrosion tests since cracking inevitably initiates at the surface where a pit or crack forms. Therefore a procedure was developed which would maintain surface uniformity among specimens used in this study.

Figure 3.2.4.2.1 - stressing jig with
"Dog-bone" and tensile rod specimens.

Tensile rods were machined to give a 30 micron surface finish. The specimens were placed in PYREX tubes with titanium sponge, and these assemblies were subjected to several cycles of vacuum followed by an inert (AR) gas purge (Figure 3.2.4.2.2). Titanium sponge was placed in the tubes to react with residual oxygen while the specimens were heat treated. The tubes were sealed under vacuum. The specimens then were heat-treated which produced a slight tarnish. They were not subjected to further surface preparation before being tested in the appropriate media.

Tensile dog-bone specimens were tested with the original 2B surface finish unmodified.

3.2.4.3 Welded Plate with Abraded Surface

The coupons described previously were fabricated from flat plate or sheet stock which was 0.125" or less in thickness. Thicker specimens were fabricated from two 4" x 12" x 5/8" pieces of 304 stainless steel plate which were butt-welded together along their lengths with 316L stainless steel welding rod. While they were being welded, the two pieces were clamped in restraint and a high heat input was used to produce weld stress and sensitization, respectively, in the metal. A current of 165 amps was used in the welding process. A total of eight welding passes was made (Figure 3.2.4.3.1). Weld stresses caused the specimens to bend after they were removed from the welding jig.

Specimens were cut from the welded plates by sectioning them perpendicularly to the weld seams to give 5/8" thick by 5/8" square bars, 8" long, which contained a transverse weld seam in the middle.

Welded specimens were ground on two edges with coarse grit grinding paper of the type usually used to remove scale from a weld. The saw used to cut specimens from the original welded plate left deep grooves on the side of each specimen. No attempt was made to alter these grooves before the specimens were tested. Finished specimens are shown in Figure 3.2.4.3.2.

Failure criteria for these specimens included crack initiation in the 10-30 mil cold-worked surface layer with crack propagation into the nonworked metal below this level.

Figure 3.2.4.2.2

Tensile rod specimens, prepared
for heat sensitization, sealed
in PYREX tube with titanium
sponge.

Figure 3.2.4.3.1
Sketch of Joint Made Between
Two 5/8"-Thick Plates of 304 SS
Butt-welded Together

The plates were clamped
in restraint while they
were welded.

Figure 3.2.4.3.2

5/8"-thick 304 SS stress specimens
with 4 types of surfaces present.

3.2.4.4 Wedge Open Loading (WOL) Stress Specimens

The wedge open loading method involves tension testing of notched specimens which have been precracked in fatigue. Load versus displacement across the notch at the edge of the specimen is recorded graphically. The load corresponding to a 2% increment of crack extension is established by a specified deviation from the linear portion of the recording. The plain strain fracture toughness, K_{IC} , is calculated from this load by equations which have been established on the basis of elastic stress analysis.

The property K_{IC} determined by this method characterizes the resistance of a material to fracture in some environment when the material contains a sharp crack under severe tensile constraint.

WOL specimens were studied because they are small and can be self-stressed easily. Eight such specimens were fabricated by Southwest Research Institute from sensitized (1200°F, 50 hours) stainless steel supplied by the Dow Chemical Company. All specimens were made and stressed according to ASTM standard E399. The stress intensity extended from 10.0 to 32.0 Ksi root-inches, where Ksi was in kilograms/in².

These specimens were exposed to the appropriate test media, evaluated, and returned to Southwest Research Institute for independent evaluation of changes in K_{IC} values.

3.2.5 Dynamic Test Loop

Cleaning solvent will be circulated through the primary system of the Dresden-1 plant by the main coolant pumps. The compatibility of these pumps with Dow NS-1 solvent at 250°F, the planned decontamination temperature, needed to be determined under dynamic conditions even though the corrosion rates of 304 and 306 stainless steels were shown to be minimal under static conditions (see Laboratory Corrosion Trial Results). The GRAPHITAR 14 bearings contained in the pump also required testing under simulated use conditions to determine their performance in Dow solvent NS-1.

Provisions were made to test galvanic couples, crevice specimens, weight loss coupons, and stress cracking specimens in the dynamic loop so that data collected from these tests could be compared with the results obtained under static conditions. Electrochemical probes also were inserted into the loop so that possibility of electrochemically monitoring the decontamination could be evaluated.

Figures 3.2.5.1 and 3.2.5.2 show a schematic diagram and a photograph, respectively, of the test loop. This simply designed loop contains a small immersion pump (P-1) which takes liquid from a 25 gallon reservoir tank (D-1). A specimen holder fits inside this tank and accommodates a number of variously shaped corrosion coupons (Figure 3.2.5.3). A single-pass, 316 stainless steel heat exchanger (E-1) maintains the temperature of the loop.

Initial thermodynamic calculations indicated that the pump would supply more energy to the loop than would be necessary to maintain the temperature at 250°F. Therefore both cooling water and steam were supplied to the heat exchanger. Steam was used to heat the loop, after which a mixture of steam and water was used to control the temperature. Electrochemical corrosion probe holders also were designed for the loop. Standard PETROLITE probes connected to a portable PETROLITE corrosion rate monitor (Figure 3.2.5.4) were used to measure corrosion. The loop was designed so that temperature and flow rate could be recorded throughout each test.

A hollow holder made from two pieces of TEFLON was designed to contain specimens for high velocity tests, as shown in Figure 3.2.5.5. Flat plate specimens were placed in one piece of the holder parallel to the direction in which a solution would flow through the holder. The two halves of the holder were joined together, and this assembly was inserted into the piping of the test loop. A tapered flow channeling device was placed upstream of the holder to direct fluid to the holder. The snug fit of the holder in the piping ensured that all solution passed through the center of the holder. The rate of flow of fluid through the holder at a given pump speed was determined by the number of specimens within the holder.

Safety devices on the loop included a pressure relief valve (PRV-1) and a flow loss control switch so that extreme temperature excursions or loss of flow would not significantly damage the loop. All metal areas that would be thermally hot when the loop was operated were insulated to prevent injury to personnel by inadvertent contact with the loop.

Figure 3.2.5.1 - Schematic Diagram of Dynamic Test Loop

- E-1 - 10 Sq. Ft., Type 316 SS
- D-1 - 25 Gallon Surge Tank
- P-1 - 3/4" x 1" Chem-Pump-G-14 Bearing Test
- PI-1 - Pressure Gauge
- FT-1 - Flow Transmitter
- TT-1 - Temperature Transmitter
- TRC-1 - Temperature Recording Controller
- FV-1, 2, 3, - Flow Valves
- PRV-1- Pressure Relief Valve

FIGURE 3.2.5.2
DYNAMIC TEST LOOP

FIGURE 3.2.5.3

Specimen holder loaded with specimens
to be placed in Dynamic Test Loop.

FIGURE 3.2.5.4

CORROSION RATE METER AND CORROSION
PROBE USED IN DYNAMIC LOOP TESTING

FIGURE 3.2.5.5

TEFLON HOLDER FOR HIGH VELOCITY TEST

3.3 LABORATORY CORROSION TRIAL RESULTS

3.3.1 Type 300 Series Stainless Steels

Type 304 stainless steel represents the largest portion of the various 300 series stainless alloys which will be exposed to Dow solvent NS-1 during the planned decontamination. Consequently, the corrosion properties of this alloy were studied in depth. In addition to 304 stainless steel, several other austenitic stainless steels (302, 303, 304L, 308, 316, and 347) were tested and will be discussed in this section.

3.3.1.1 Summary of Results

Type 304 stainless steel when annealed or extensively cold-worked, had low general corrosion rates of 1-2 mpy when exposed to Dow solvent NS-1 at up to 275°F and 300 hours, and when unsensitized was not subject to localized forms of corrosion. The other 300 series alloys performed similarly, and exhibited corrosion rates of 1-4 mpy.

Pitting and crevice attack was not initiated on the crevice specimens tested. Galvanic coupling of the 300 series stainless alloys to several other alloys caused no unacceptable corrosion effects. Exposure of 300 series alloys to Dow solvent NS-1 did not produce stress corrosion, even after the alloys had been extensively cold-worked.

Corrosive attack of unsensitized 300 series stainless steels in Dresden-1 is not anticipated, therefore, as a result of decontaminating the primary reactor with Dow solvent NS-1.

The 304 stainless steel cladding on the Dresden-1 reactor pressure vessel was exposed to sensitization-producing temperatures when the cladding was stress relieved at the time it was fabricated. For this reason, specimens from a number of different heats of 304 stainless steel which had been sensitized by post-weld heat treatment (PWHT) were comprehensively tested in Dow solvent NS-1. The severity of the sensitizations exceeded that actually used when the reactor was fabricated. PWHT stress relief in pressure vessels usually is achieved by maintaining the parts at 1000-1150°F for one hour, plus an additional heat treatment for one to two hours per inch of metal thickness. Most of the specimens used in this corrosion study were sensitized at 1200°F for 50 hours, thus producing more severe carbide precipitation and susceptibility to intergranular stress corrosion cracking than actually would be found in Dresden-1.

General corrosion rates of sensitized 304 stainless steel ranged from <1 to 5 mpy, and were not significantly different from those of the unsensitized alloy. Corrosion rates of other alloys galvanically coupled to sensitized 304 stainless steel also were low with no susceptibility to pitting corrosion or crevice attack noted as a result of testing in NS-1 solvent at temperatures to 275°F.

The IGSCC tendency of sensitized and cold-worked 304 stainless steel was evaluated in depth for several hundred specimens which varied in type from single and double two-stage U-bends to more complex tensile rod specimens. In addition, 1/8" to 5/8"-thick weld specimens of 304 stainless steel which contained different types of fabrication stresses were evaluated for stress corrosion behavior.

Intergranular stress corrosion cracking was not encountered on sensitized 304 stainless steel exposed to Dow solvent NS-1 under the conditions planned for the cleaning of Dresden-1. However, sensitized 304 stainless steel which had been stressed beyond its yield point did become susceptible to IGSCC when exposed to either deionized water or Dow solvent NS-1.

No significant effect on the cracking susceptibility of the alloy was found in environmental modification studies with static vs rocking bomb trials, high vs low oxygen concentrations, inert gas (N₂) vs air padding, or in the presence of an oxidizing agent (hydrogen peroxide) vs a reducing agent (hydrazine).

No IGSCC was observed on specimens from any heat of 304 stainless steel when the specimens were sensitized after they were stressed, the condition expected for any small areas of sensitized 304 steel in Dresden-1.

These results indicated that decontamination of Dresden-1 with Dow solvent NS-1 will not result in conditions more corrosive to sensitized 304 stainless steel than encountered when deionized water is used.

Individual corrosion test data sheets for 304 stainless steel and the other 300 series alloys are given in section 3.10.5.

3.3.1.2 General Corrosion

Weight loss data for austenitic stainless steels indicated that little corrosion of the metals will occur when Dresden-1 is cleaned with Dow solvent NS-1. Type 300

series alloys exposed 16 hours to simulated used Dow solvent NS-1 corroded at <2 mpy as shown in Table 3.3.1.2.1. Corrosion rates under these conditions remained constant after 100 hours, an indication that the corrosion mechanism did not change with time. Coupons of 304 and 304L alloys, which were heated in air to induce sensitization after 100 hours exhibited weight losses which were due chiefly to removal of heat scale by the solvent. No pitting, crevice, or other localized attack was noted on 300 series alloys at temperatures up to 275°F, which is 25°F higher than the planned decontamination temperature, when the specimens contained sheared edges and extensively cold-worked areas.

Specimens and the solvent were tested in both glass and TEFLON containers. Additional tests showed that the corrosion rate of 304L stainless steel did not appear to be a function of the test container:

<u>Container</u>	<u>CR(mpy) of 304L SS in NS-1 solvent (100 hr, 250°F)</u>
glass	0.18, 0.2
TEFLON	0.1, 0.4, 0.68

FIGURE 3.3.1.2

As received 2B surface finish on 304L SS compared
to grit blasted surface of 304L SS.

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Table 3.3.1.2.2

General Corrosion Rates Of 304 SS and 304L SS
Obtained In Vapor Phase At 250°F
In Air-Saturated Dow Solvent NS-1

<u>Metal</u>	<u>16 hour CR(mpy)</u>	<u>100 hour CR(mpy)</u>
304 in Vapor		0.7, 0.5
304 Submerged		0.9, 0.4
304L in Vapor	1.5, 1.7	
304L Submerged	1.3, 1.0	

At the standard use concentration standard, the corrosion inhibitor in Dow solvent NS-1 functioned adequately, as evidenced by the low general corrosion rates observed for 304 stainless steel specimens. However, variable corrosion results were obtained at 275°F, a temperature 25°F higher than that planned for the actual decontamination. Some specimens corroded at <1 mpy in NS-1 solvent at 275°F whereas other specimens from the same heat of stainless steel corroded at 50 mpy (Table 3.3.1.2.3).

The corrosion rates obtained at 275°F were not excessive although they were greater than expected and were non-uniform. These results suggested that the standard concentration of inhibitor in NS-1 solvent was ineffective at 275°F. Increasing the concentration of inhibitor to 2x standard, see Table 3.3.1.2.3, improved the uniformity of the corrosion performance of the solvent. Additional evaluation of the inhibitor concentration is described in the section entitled Effect of Inhibitor Concentration.

Table 3.3.1.2.3

High Temperature Corrosion Rate For
Austenitic Stainless Steels
(Mils/Year)
Exposed 112 Hr to Dow Solvent NS-1

<u>Metal</u>	<u>250°F^a</u> <u>NS-1</u>	<u>275°F^a</u> <u>NS-1</u>	<u>275°F^b</u> <u>NS-1</u>
SS 304 Heat I	0.45	82	Same specimen in both runs
SS 304 Heat II		0.34 0.50	
SS 304 Heat III		0.29 0.32 0.27	
SS 304 Heat IV		0.35	0.17
SS 304 Heat VI	0.18 0.09 0.15	33, 43, 0.54 0.41 31, 0.19, 0.19	0.11 0.22 0.22
Sens. SS 304 Heat II	2.8 2.5	71 83	2.6 3.0 2.5
Sens. SS 304 Heat III	2.8 3.7 1.9	64 73 76	3.4 3.7 3.3
Sens. SS 304 Heat IV (descaled)	47 2.6	2.6, 84 69 83, 90	2.8 2.8 3.1
SS 304L Heat I		0.41 0.38 0.59	
SS 304 Heat II	0.15 0.15	58 24	0.26 0.22 0.24

^aWith standard inhibitor concentration

^bWith 2x standard inhibitor concentration

Table 3.3.1.2.3 (Continued)

	<u>250°F</u> <u>NS-1^a</u>	<u>275°F</u> <u>NS-1^a</u>	<u>275°F</u> <u>NS-1^b</u>
SS 304L Heat IV	0.10 0.18 0.26	38, 0.19, 0.30 7.7, 21 0.51, 2.3, 59	0.15 0.26 0.14
Sens. SS 304L Heat II	40	38 62 74	1.9 1.7 15
Sens. SS 304L Heat IV	5.4 1.9	63 37 59	2.2 2.6 2.9
SS 316	0.62 0.63	15, 0.83 0.65, 20 19, 0.54, 7.7	0.68 0.37 0.44

All corrosion rates were taken on single U-bends under static conditions. No cracking was observed on any U-bend.

^aWith standard inhibitor concentration

^bWith 2x standard inhibitor concentration

3.3.1.3 Galvanic Corrosion

Bimetallic specimens based on couples of 1020 carbon steel with 304 stainless steel were tested in Dow solvent NS-1 as shown in Table 3.3.1.3.1. Specimens in which the 1020 carbon steel was coupled to 304 stainless steel at an area ratio of 1:1 had similar corrosion rates regardless of the type of coupling: e.g., 1020 carbon steel in welded, bolted, and rubber-banded couples had corrosion rates of 114-258 mpy, 214-228 mpy, and 63-204 mpy, respectively, when the specimens were exposed to NS-1 solvent for 112 hours at 250°F. The similarity of these rates indicates that most of the corrosion arose from the bimetallic character of the specimens, and that crevice effects from the rubber-banded samples were of secondary importance.

Displacing air with nitrogen did not decrease the galvanic attack. Specimens of 1020 carbon steel rubber-banded to 304 stainless steel corroded at a rate of 166-188 mpy in air after 16 hours, whereas under nitrogen rubber-banded and bolted 1020 carbon steel samples corroded at 173-187 mpy.

When the area ratio of 1020 carbon steel coupled to 304L stainless steel was increased from 1:1 to 1:10, respectively, the corrosion rate of the 1020 steel increased from an average of 165 mpy to approximately 750 mpy. When the area ratio was increased to 1:30, the corrosion rate of the 1020 member increased to 1550 mpy; i.e., the galvanic corrosion rate doubled with a three-fold increase in area ratio. The 1:30 area ratio was intended to simulate the condition which exists in the 304 stainless steel cladding area at Dresden-1. Although these corrosion rates were significant, they are not expected to be unacceptable since significant metal loss probably will not occur during the proposed cleaning time of 100 hours.

In summary, coupling 1020 carbon steel to 304 stainless steel, regardless of the method by which the metals were coupled, increased the corrosion rate of the carbon steel by an order of magnitude but did not significantly affect the corrosion rate of 304 stainless steel when the metals were exposed to NS-1 solvent. As the area ratio of stainless to carbon steel increased, the corrosion rate of the carbon steel increased correspondingly, but the corrosion mechanism remained the same.

Table 3.3.1.3.1 (page 1 of 3)

Galvanic Corrosion Data on Banded and Bolted Couples of
Selected Alloys at 250°F in Air-Saturated Dow Solvent NS-1

<u>Specimen^a</u>	<u>Type of Couple</u>	<u>16 hour CR(mpy)^b</u>	<u>112 hour CR(mpy)^b</u>
1020 to 304L	welded	138, 174, 127	120, 114, 167, 131 202, 258, 176
1020 to 304L	banded	188, 175, 166 1.3, 1.0, 1.4	63, 39, 156, 204 1.4, 5.1, 4.3, 2.0
1020 to 304L (N ₂ Sat'd)	banded	187 3.0	
1020 to 304L	bolted with spacer	216, 212 1.3, 0.9	214, 228
1020 to 304L (N ₂ Sat'd)	bolted with spacer	173 2.6	
1020 to 304L (10:1 area ratio)	banded	38, 43, 45 0.45, 25, 1.5	
1020 to 304L (1:10 area ratio)	banded	799, 726 0.71, 0.53	771 2.4
1020 to 304L (1:30 area ratio)	banded	1550 0.50	
1020 to 304L	banded	159, 164 0.80, 0.12	
1020 to 316	banded	160 0.30	
1020	uncoupled	16, 25, 25, 25	

Table 3.3.1.3.1 (Continued)
page 2 of 3

<u>Specimen^a</u>	<u>Type of Couple</u>	<u>16 hour CR(mpy)^b</u>	<u>112 hour CR(mpy)^b</u>
1020 to 304 SS	banded	159, 164 0.80, 1.2	188, 175, 166 1.3, 1.0, 1.4
1020 to 316 SS	banded	160 0.30	
1010 welded to itself coupled to 304L	banded		166, 212, 298 8.5, 7.1, 5.8
1010 welded to itself coupled to 304L	bolting		424, 263 9.0, 8.2
2-1/4 Cr - 1 Mo to 304	banded		475 16
2-1/4 Cr - 1 Mo to 304	bolting		440 1.8
INCONEL ^c - 600 to 304	banded		0.4 0.1
INCONEL - 600 to 304	bolting		1.2 0.1
17-4 P.H. to 304	banded		0.2 0.1
17-4 P.H. to 304	bolting		0.3 0.1
17-4 P.H. to 304	welded	1.6	

Table 3.3.1.3.1 (Continued)
page 3 of 3

<u>Specimen^a</u>	<u>Type of Couple</u>	<u>16 hour CR(mpy)^b</u>	<u>112 hour CR(mpy)^b</u>
STELLITE ^d 6 to 304	bolted		0.1 0.3
Cr 122 to 347	banded		2.6 0.3
Cr 122 to 347	bolted		3.1 0.4
70-30 Cu-Ni 304L	banded	6.4 0.70	3.1 0.41
Al 1100 to 304L	bolted	148, 162 0.49, 0.37	
405 SS to 304L SS	banded	2.3, 2.1 0.36, 0.84	
410 SS to 304L SS	banded	3.3 0.31	
4142 to 304L SS	banded	191 2.3	258, 243
B-1213 to 304L SS	banded	174 1.0	398, 124 2.4, 5.5

^aThe area ratio was 1:1 unless otherwise noted.

^bWeight loss (corrosion rate) on welded couples was assumed to occur only on the anodic member. Otherwise, the corrosion rate applies to the specimen material appearing on the same line of print.

^cTrademark of Huntington Alloys, Inc.

^dTrademark of Stellite Division, Cabot Corporation.

Welded specimens of 1010 carbon steel, in which approximately 1/8 of the surface was weld area, were coupled to 304L stainless steel. Corrosion rates of these couples in NS-1 solvent varied from 166-424 mpy; attack of the weld was not detected.

Low alloy steels such as 2-1/4 Cr - 1 Mo, 4142, and B1213 exhibited higher corrosion rates than ordinary carbon steels when coupled to 304 or 304L stainless steels, whereas the 304 alloy corroded at generally constant rates of 2 mpy or less regardless of the alloy to which it was coupled. Corrosion rates of the 2-1/4 Cr - 1 Mo alloy coupled to 304L stainless steel varied from 440-475 mpy. The 4142 alloy coupled to 304L stainless steel corroded at a rate of about 250 mpy. All attack on low alloy and carbon steels was uniform, with no pitting, crevice corrosion, or excessive corrosion rates found as a result of Dow solvent NS-1.

Aluminum alloy 1100 bolted to 304L stainless steel corroded at 148-162 mpy in NS-1 solvent after 16 hours, a rate similar to that found for carbon steel.

Copper 122 and copper nickel 70-30, as bimetallic couples with 347 and 304L stainless steels, respectively, corroded at rates of 3-4 mpy after 100 hours in NS-1 solvent, with the stainless alloys corroding at <1 mpy.

When 410 and 405 stainless alloys were coupled to 304L stainless steel, the 410 alloy corroded at a rate of 3.3 mpy after 16 hours in NS-1 solvent, whereas the 405 alloy corroded at 2.1-2.3 mpy. The corrosion rates exhibited by 304L remained at <1 mpy for both couples.

In couples with 304 stainless steel, INCONEL^a 600 and 17-4 precipitation hardened stainless steel gave low corrosion rates in NS-1 solvent, although both alloys were anodic toward 304 stainless steel. In only one specimen did the corrosion rate exceed 1 mpy; i.e., in INCONEL bolted to 304 stainless steel at a 1:1 ratio, and exposed to NS-1 solvent for 100 hours, the corrosion rate was 1.2 mpy.

Only one of the alloys coupled to the 300 series stainless alloys was cathodic to 304 stainless steel, namely STELLITE^b 6 which corroded at 0.1 mpy after 100 hours in NS-1 solvent. The 304 alloy remained passive and corroded at 0.3 mpy.

^aTrademark of Huntington Alloys, Inc.

^bTrademark of Stellite Division, Cabot Corporation

In summary, 304 and 304L stainless steels, which were chosen to represent the 300 series alloys, did not show excessive galvanic coupling effects, and in general corroded at rates of <5 mpy when exposed to Dow solvent NS-1. No pitting or crevice attack on the 304 alloy was observed, and all metals tested, except STELLITE 6, were anodic to the 304 alloy.

Some alloys which did exhibit galvanic coupling, namely carbon and low alloy steels, were subjected to additional galvanic tests. Galvanic corrosion data for SA516 Gr70 and SA36 exposed to NS-1 solvent are shown in Table 3.3.1.3.2. Both of these steel alloys are representative of the structural steel in Dresden-1, and would be exposed to Dow solvent NS-1 only if a cladding failure occurred. The bolting on rubber-banded specimens did not influence the corrosion rates, an indication that crevice effects were not important. These structural steels behaved similarly to 1020 carbon steel and the low alloy steels, and corroded at rates of 400-500 mpy when coupled to 304L stainless steel in a 1:1 area ratio. When the area ratio was changed to 1:10 in favor of 304L stainless steel, the corrosion rate of SA516 Gr70 increased to 1600-2000 mpy. However, the attack was uniform and was not localized on any given part of the specimens. Corrosion rates of 304L stainless steel increased slightly from those observed with 1020 carbon steel and some of the other low alloy steels, and were found to be 1 mpy, or less, to 16 mpy. These higher corrosion rates, relative to those of other steel alloys, apparently were caused by dissolved corrosion products. Corrosion of the stainless steel was uniform rather than localized.

The galvanic corrosion rates shown in Table 3.3.1.3.1 and Table 3.3.1.3.2 were measured under static conditions. Bimetallic couples of 304 stainless steel with 1020 carbon steel and 17-4 precipitation hardened (P.H.) stainless steel also were subjected to dynamic tests to determine the influence of solvent velocity on corrosion of these metals in NS-1 solvent. The data below show the galvanic corrosion effects under dynamic conditions:

Specimen ^a	CR(mpy) ^c
304 SS welded to 102 ^b	96, 101
304 SS (sen.) welded to 1020 ^b	102, 97
17-4 (P.H.) welded to 304 SS ^b	0.18, 0.53

^a0.12 wt%, 0.065 wt% Ni⁺² added as contaminants

^banodic member of couple

^c100 hrs, 250°F, NS-1 solvent saturated with air; corrosion rates were calculated by assuming all wt loss occurred on anodic member.

Table 3.3.1.3.2

Galvanic Corrosion Data on Banded and Bolted Couples
of Two Low Alloy Steels in Air-Saturated Dow Solvent
NS-1 at 250°F for 112 Hours

<u>Specimen</u>	<u>Area Ratio</u>	<u>Corrosion Rate (mpy)</u>
SA 516 Gr70 banded to 304L	1:1	489 2.1
SA 516 Gr70 bolted to 304L	1:1	533 1.3
SA 516 Gr70 banded to 304L	1:10	1838, 2063, 1889, 1889 1833, 1613, 1964, 1807 9.8, 7.0, 1.9, 4.6, 2.1 3.4, 2.9, 5.5
SA 516 Gr70 welded to itself, banded to 304L	1:1	418, 400 3.9, 0.12
SA 516 Gr70 welded to itself, bolted to 304L	1:1	493, 455, 578 2.5, 4.8, 5.2
SA 516 Gr70 banded to sens. 304	1:1	489 1.5
SA 516 Gr70 bolted to sens. 304	1:1	521 2.1
SA 36 banded to 304L	1:1	453 3.5
SA 36 bolted to 304L	1:1	511 2.4
SA 36 welded to itself, banded to 304L	1:1	493 16
SA 36 welded to itself, bolted to 304L	1:1	500, 467, 413 5.0, 6.1, 5.6

Note: The corrosion rates listed are associated with the specimen material which appears on the same line of print.

Specimens of 304 stainless steel, and of 304 stainless that had been sensitized for 50 hours at 1200°F, were exposed to simulated used Dow solvent NS-1 for 100 hours in a shaker over which revolved 3x/second. Welded couples were tested, in which the loss was assumed to have occurred only on the anodic member of the couple. These alloys were considered to represent two extremes of possible galvanic couples. Corrosion rates of 1020 carbon steel and 17-4 precipitation hardened stainless steel appeared to be somewhat lower under dynamic conditions than under static conditions. No weld attack was observed.

Previous corrosion tests did not include evaluation of galvanic couples under stress; thus a series of dissimilar double U-bends were constructed with U-bends of 304 stainless steel forming the outer bend of the couple. Area ratios were maintained at 5:1 in favor of the 304 alloy. The corrosion rates of 1020 carbon steel in such stressed specimens were 193-220, and thus were not significantly higher than rates obtained for unstressed specimens (63-204 mpy) when both types of specimens were exposed to NS-1 solvent for 112 hours.

As shown in Table 3.3.1.3.3, the corrosion rate of 405 and 410 stainless steel alloys exposed to NS-1 solvent did not appear to be affected by stress or increased 304 stainless steel areas at an area ratio of 5:1 in favor of the 304 alloy. The 405 and 410 stainless steel alloys corroded at a rate of 3.1-5.7 mpy under stress, and corroded at 2.1-2.3 mpy without stress and at an area ratio of 1:1. Stress effects were not observed on a couple of 304 stainless to 405 or 410 stainless steel. Specimens of 446 stainless steel had low galvanic corrosion rates (<1 mpy) even when ferric and nickelous ions were present in the solvent.

When coupled to 304 stainless steel at a 1:5 area ratio, B1213 showed evidence of stress effects, and had a corrosion rate of 393-553 mpy compared to a corrosion rate of 124-398 mpy observed in the absence of stress. This increased corrosion rate can be attributed partly to the increased area ratio of the cathodic stainless surface and to the ferric and nickelous ion contaminants in solution. No stress cracking was observed but stress etching was evident on the B1213 specimens. Sulfur stringers in this steel were anodic to the surrounding matrix and were preferentially attacked. Metallographic data for stressed B1213 are given Appendix I (test LIV-13-16).

Table 3.3.1.3.3

Galvanic Corrosion Data on Stressed Double U-Bends
Exposed to Dow Solvent NS-1 at 250°F for 100 Hours
and then to Deionized Water at 500°F for 200 Hours

<u>Specimen</u>	<u>Contaminants</u>	<u>Area Ratio</u>	<u>CR(mpy) of Alloy Coupled to 304</u>
405 inner	0.12 wt% Fe ⁺³	1:5	3.3, 3.1, 3.1, 3.7
304 outer	0.065 wt% Ni ⁺²		(No cracking)
410 SS inner	"	"	5.7, 4.9, 5.3, 5.5
304 outer	"		(No cracking)
446 inner	"	"	0.1, 0.1, 0.1, 0.1
304 outer	"		(No cracking)
1020 inner	"	"	206, 193, 200, 220
304 outer	"		(No cracking)
B1213 inner	"	"	553, 467, 481, 393
304 outer	"		(No cracking)

U-Bend couples were made by placing alloy U-bend inside a 304 U-bend.

Stress cracking occurred on the sensitized inner U-bend of specimens in which 304 stainless steel sensitized at 1200°F for 50 hours was coupled galvanically to non-sensitized 304 stainless and exposed to solvent which contained 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} . The heat scale which was left intact appeared to contribute to the significant weight losses observed for the sensitized specimens subjected to cleaning. These results are discussed further in section 3.3.1.5 on stress corrosion.

In summary, 304 stainless steel was cathodic when coupled to other alloys, except STELLITE 6, and did not exhibit decreased passivity in NS-1 solvent when tested as the cathodic member of such bimetallic couples. Weight losses were minimal, and corrosion was uniform at rates of <5 mpy. Carbon and low alloy steels were strongly anodic to 304 stainless steel. The other 300 series alloys are expected to perform similarly, and therefore were not screened.

3.3.1.4 Crevice Corrosion

The 300 series stainless alloys are noted for their general corrosion resistance which is attributed to the formation of oxidatively-produced surface films which inhibit dissolution of metal species. Chemical or mechanical disruption of this passive film allows the alloy to undergo localized crevice or pitting attack at the damaged areas. These types of corrosion can occur rapidly and adversely affect the performance of the alloys. Various tests therefore were performed to determine the crevice corrosion behavior of series 300 stainless steel alloys in Dow solvent NS-1.

Electrochemical Crevice Tests

Both cursory and in-depth electrochemical studies were carried out on Dow solvent NS-1. The initial cursory studies of initiation of crevice attack on selected alloys were performed within the Dow Chemical Company, whereas the in-depth studies of the crevice and stress corrosion behavior of 304 stainless steel were carried out at The Ohio State University by Professor R. W. Staehle and Professor A. K. Agrawal.

The results of the pitting and crevice initiation tests will be described briefly; the Staehle report is included in the Appendix.

Tests on initiation of crevice attack were carried out by using cycle potentiodynamic polarization to increase the critical pitting potential (E_{cpp}) of the following

alloys: 304 stainless steel, 304 stainless steel sensitized 50 hours at 1200°F, 17-4 precipitation hardened stainless steel, 410 stainless steel, 308 stainless steel STELLITE 6, and 1020 carbon steel.

In this test, the anodic potential of an alloy is slowly increased to higher values until the passive film ruptures and localized attack ensues at the rupture. Determining the displacement of the E_{ocp} at which film rupture occurs, from the open circuit potential of the alloy, gives some indication of the resistance of the alloy to pitting and crevice corrosion. Usually hysteresis is associated with the reverse polarization scan as the alloy returns to its original open circuit potential (E_{ocp}) after the desired anodic value is reached. If the return scan exhibits values which are more positive than shown in the original scan, the alloy can be expected to show little tendency for propagation of pitting or crevice attack even after initiation of the attack has occurred. However, if the reverse polarization scan exhibits values which are more anodic (negative) than shown by the original scan, the alloy can be expected to undergo possibly severe crevice and pitting attack after such types of attack have initiated.

After initiation of crevice or pitting attack on an alloy, the solution within a pit will exhibit a decrease in pH to 1-3 because of aqueous hydrolysis of the corroding metal within the pit or crevice. As the pH decreases, the corrosion rate within the pit accelerates until reaction products suppress or stop the reaction.

An extreme pitting or crevice corrosion situation was simulated for testing the effects of Dow solvent NS-1 on the alloys described previously in this section. Each specimen was equipped with a snug-fitting O-ring which produced a crevice. The pH of the solvent was decreased to one with concentrated sulfuric acid; 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} were added to give a solution which simulated that found within a crevice.

Each specimen was allowed to equilibrate with a fresh solution of NS-1 solvent for 24 hours, and then was polarized anodically at 10 millivolts/minute to 700 mv above the open circuit potential of the specimen, followed by a reverse scan to the original open circuit potential (Figures 3.3.1.4.1 through 3.3.1.4.7). The area under the O-ring around each polarized specimen was examined for initiation of crevice attack.

The reverse polarization scans of the alloys intersected the potential axis at values which were more positive than the original E_{ocp} , except for STELLITE 6 which exhibited a reverse scan that intersected the original E_{ocp} value.

The polarization curves indicate that the specimens were passive in the test solution, with the exception of 1020 carbon steel and 410 stainless steel which underwent attack in the solvent at pH 1. The attack on these specimens was uniform, and was not localized beneath the O-ring. The alloys which were passive in pH-adjusted solvent showed no indication of pitting or crevice attack. None of the alloys exhibited a sudden increase in current with small changes in potential, an indication that rupture of the passive film and subsequent localized attack did not occur.

Initiation of crevice and pitting attack therefore was not found when these alloys were exposed to Dow solvent NS-1. The tests were carried out at 80°C, a temperature lower than that planned for use during the actual cleaning of Dresden-1, because of the unavailability of a reference electrode to monitor potentials at higher temperatures. Electrochemical tests at higher temperatures were carried out in autoclaves at The Ohio State University.

-63-
Current Density (I) in Microamps Per Cm²

CONDITIONS:

Temperature . . . 81°C
Sweep 10 mv/min
OCP +146
Reference SCE
pH ~1.0
Equilibration
Time ~64 hours

Figure 3.3.1.4.2

Electrochemical Crevice Test
for 304 Stainless Steel

-L9-
Current Density (I) in Microamps per CM²

Figure 3.3.1.4.7

Electrochemical Crevice Test
for 17-4 P.H. Stainless

CONDITIONS:

Temperature . . . 80°C
Sweep 10 mv/min
OCP +171 mv
Reference SCE
pH ~1.0
Equilibration
Time ~19 hours

Crevice Test with pH Electrode

Solution chemistry plays an important role in the mechanism of crevice corrosion. Acidification resulting from the hydrolysis of metal ions trapped in a crevice can activate crevice corrosion on an initially passive surface. Marek and Hockman and others obtained useful data on localized corrosion processes by studying macrocells which simulated crevice conditions on a large scale.

In the present study, a crevice was simulated on 304 stainless steel by drilling a 2" diameter by 1.5"-thick rod of the alloy with a cavity that conformed closely in shape to that of a pH electrode which was inserted into the cavity. An insulated connection was attached to the outside of the specimen. This arrangement allowed measurement of potentials inside the crevice with the reference electrode junction, and measurement of potentials outside the crevice with the insulated connection. (Figure 3.3.1.4.8).

The machined specimen of 304 stainless steel was sensitized at 1200°F for 50 hours and was allowed to cool in the furnace. This sensitized alloy was exposed to Dow solvent NS-1 at 80°C for 100 hours under static conditions.

Initially, both the solvent in the simulated crevice and the bulk of the solvent around the specimen had pH values of 3.60. The pH values increased to 3.80 and 3.75, respectively, at the end of the 100 hour test. The potentials (measured relative to an Ag/AgCl reference) were +0.11 mv inside the crevice and +0.93 mv outside the crevice.

The electrode assembly was carefully removed from the solvent to ensure that solvent was retained within the crevice. Iron and nickel concentrations were analyzed:

	concentration (ppm)	
	iron	nickel
Bulk solvent	2.5	3
Solvent in crevice	2.5	7

These results indicate that significant crevice attack will not occur on sensitized 304 stainless steel exposed to Dow solvent NS-1 during a 100 hour cleaning cycle.

Figure 3.3.1.4.8

Electrode assembly for crevice pH and
potential measurements on 304 SS

Visual inspection of the machined crevice further indicated localized attack had not occurred. Tests should be made to evaluate the performance of NS-1 under a wider range of conditions.

Stressed Crevice Test

Double U-bends of 304 stainless steel were exposed to simulated used Dow solvent NS-1 in order to test the effects of stress and contaminants on initiation of crevice attack. U-Bend specimens of 3/4" and 2"-wide alloy were sensitized for 50 hours at 1200°F and were allowed to cool in the furnace, after which they were stressed. The 2"-wide, double U-bends were intended to simulate a long thin crevice. All U-bends were descaled.

Eight 3/4"-wide U-bends were exposed for 200 hours at 250°F to NS-1 solvent saturated with air and Fe_2O_4 to simulate a stressed crevice exposed to scale-saturated solvent. Ten 2"-wide U-bends were exposed for 100 hours at 250°F to NS-1 solvent saturated with air and with 0.12 wt% Fe^{+3} and 0.65 wt% Ni^{+2} added to simulate a stressed crevice exposed to used solvent. Both types of specimens were transferred unrinsed to deoxygenated (<0.3 ppm O_2) deionized water and exposed to this media at 500°F for 200 hours.

That no stress corrosion was detected on any of these specimens, either visually or by metallographic analysis, was a further indication that 304 stainless steel crevices were not attacked by Dow solvent NS-1. Testing the U-bends at 500°F, a temperature above which the solvent decomposes, indicated that solvent degradation products did not initiate stress attack in a 304 stainless steel crevice in the presence of scale, Fe^{+3} , or Ni^{+2} .

A technique reported by Larrick and Weed⁴⁷ who used fitted metal tubing specimens to study corrosion of stressed crevices was adapted to the present study. Fitted 304 stainless steel tubing which contained extensive cold-worked areas as a result of having been machine fabricated was sensitized at 1200°F for 50 hours (Figure 3.3.1.4.9). Four such specimens were exposed for 100 hours at 250°F, the temperature planned for cleaning Dresden-1, to Dow solvent NS-1 saturated with air and contaminated with 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} . Each piece of tubing and its associated fitting was assembled under the solvent before each test to ensure that solvent was trapped in the simulated crevice. Figure 3.2.3.3.1 shows the two types of simulated crevices present on each specimen: one which allowed diffusion of solvent, and the other which completely sequestered the solvent.

Figure 3.3.1.4.9

Fitted tubing specimen of 304 SS exposed to simulated used Dow solvent NS-1 for 100 hours at 250°F and then to deionized, deoxygenated water at 500°F for 100 hours.

In subsequent tests, each specimen was heated for 100 hours at 550°F in an autoclave which contained deoxygenated, deionized water in order to decompose solvent trapped in the crevices. Localized attack was not detected on the specimens after they were subjected to this simulated hydro-test.

This test substantiated earlier findings that crevice attack is not initiated by Dow solvent NS-1 on sensitized 304 stainless steel under stress. Additionally, decomposition products from the solvent did not appear to aggravate a pre-existing crevice condition.

In summary, crevice attack is not expected to occur on 300 series alloys exposed to Dow solvent NS-1 under conditions planned for the decontamination of Dresden-1, as evidenced by the corrosion behavior of 304 stainless steel. No significant attack occurred in electrochemical tests nor when the solvent was decomposed within a pre-existing crevice. The solvent also did not initiate crevice attack on 304 stainless steel.

These results are in agreement with those of Professor Roger Staehle of The Ohio State University, and support the tenet that decontamination of the primary reactor at Dresden-1 with Dow solvent will not result in crevice corrosion of 304 stainless steel.

3.3.1.5 Stress Corrosion

Assessing the stress corrosion behavior of alloys in solvent NS-1 is not straightforward because of the lack of acceptable standardized methods. The reasons for conducting this stress corrosion study therefore dictated the approach which was taken.

The purpose of this study was to determine the stress corrosion susceptibilities of alloys when they were exposed to Dow solvent NS-1 under the conditions planned for the decontamination of Dresden-1. U-Bend specimens were used to evaluate the effects of heat treatment and other variables on the performance of the alloys. Alloys which exhibited potentially unacceptable corrosion performance in U-bend tests then could be studied more thoroughly in other tests.

The exact types of stress which will be encountered during the actual cleaning are unknown. The U-bend design was chosen because it subjects an alloy to the severest condition available for smooth stress corrosion test specimens. Such a severe stress condition is highly unlikely for any of the alloys to be exposed to the Dow solvent. The existence of a variety of stresses in a

U-bend makes it useful for detecting differences between the stress cracking resistances, in the same environment, of different metals or of specimens of a single metal under different metallurgical conditions.

Two-stage single and double U-bend specimens were used to evaluate the stress corrosion cracking characteristics of highly sensitized 304 stainless steel in Dow solvent NS-1. Specimens from five different heats of the alloy were tested. Specimens were sensitized at 1200°F for 50 hours in air, and were allowed to cool in the furnace. The sensitized specimens were fabricated into U-bends, placed in TEFLON lined spools, and tested under static conditions for 112 hours at 250°F in NS-1 solvent which initially was saturated with air. The air in the solvent was not replenished once the containers were sealed and the test started.

No detectable stress corrosion occurred on 304 or 304L stainless steel U-bends, either sensitized or unsensitized, or on 316 stainless steel U-bends at temperatures up to 275°F and for times up to 300 hours. Highly sensitized 304 stainless steel did stress crack in NS-1 solvent, however, if heat treatment scale was present or if the solvent contained added ferric and nickelous ions. The most severe cracking occurred when both of these were present (Table 3.3.1.5.1).

Approximately one-third of the specimens was tested in fresh NS-1 solvent. Another one-third was exposed to simulated used solvent, i.e., NS-1 to which 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} had been added. The remaining one-third of the specimens were descaled in NS-1 solvent under reflux conditions for 12 hours, after they were sensitized but before they were formed into U-bends. About one-half of the descaled specimens was exposed to fresh NS-1 solvent, and the other half to simulated used solvent.

Stress cracking of highly sensitized 304 stainless steel tested at 250°F for 112 hours under static conditions occurred as shown in Table 3.3.1.5.1. From the table it is seen that:

1. 12% of the sensitized heat-scaled specimens (two single U-bends and five double U-bends) exhibited intergranular cracking when exposed to fresh NS-1 solvent.
2. 73% (55 out of 75) of the sensitized heat-scaled specimens underwent intergranular stress cracking when exposed to simulated used NS-1 solvent; and

Table 3.3.1.5.1

Effect of Heat Treatment on U-Bends
Exposed to Used Dow Solvent NS-1

Total number of single U-bends 58

Total number of double U-bends 157

Total number of cracked U-bends

Single 6

Double 72

Conditions which caused cracking:

Presence of heat treatment scale	No. Tested	No. of cracked
Single	19	2
Double	15	5

Presence of heat treatment scale
with 0.12 wt% Fe^{+3} + 0.065 wt% Ni^{+2}
Added to solvent

Single	20	4
Double	55	51

No heat treatment scale but 0.12 wt%
 Fe^{+3} + 0.065 wt% Ni^{+2} added to
solvent

Single	19	0
Double	52	15

(All cracks were intergranular)

Conditions (static):

Temperature: 250°F

Time: 112 hours

Solvent: initially air-saturated (no replenishment)

Specimens: sensitized at 1200°F for 50 hrs, then
U-bent.

Note: Where cracking occurred it was IGSCC.

3. 21% (15 out of 71) of the double U-bend sensitized specimens which were descaled in NS-1 solvent after heat treatment and prior to U-bend formation underwent intergranular stress corrosion failure in simulated used NS-1 solvent; none of the single U-bends failed this test.

For the double U-bend specimens the superposition of the crevice conditions onto the elastic and plastic strains present in the single U-bend specimens represents a more severe test condition. This severe test condition was the cause of the resulting higher percentage of stress corrosion failures for the double U-bend specimens of 304 stainless steel (as compared to the single U-bend specimens).

All cracked specimens were examined with a stereo microscope, and several were mounted and examined metallographically. These examinations showed that all cracking was intergranular, as expected, since the stainless steel had been sensitized for 50 hours at 1200°F.

Cracking occurred in specimens from all five heats of 304 stainless steel, but the most significant amount occurred in samples from heat #3. The increased susceptibility of specimens from heat #3 to intergranular cracking apparently resulted from the way in which the coupons were cut from the bulk sheet. In order to provide increased susceptibility to IGSSC the coupons intentionally were cut transversely instead of parallel to the direction in which the original sheet was rolled. Each heat was similar in composition, although some contained more carbon than others.

Additional specimens from the same heat which produced specimens that cracked were tested in deionized water under conditions identical to those which produced IGSSC in Dow solvent NS-1. These stress corrosion results, shown in Table 3.3.1.5.2, indicate that cracking appeared to be more severe in deionized water than in Dow solvent NS-1.

Six specimens were tested under each of the three types of conditions. Two of six specimens underwent IGSSC when they were tested with intact heat treatment films. Six out of six heat-scaled specimens and two out of six descaled specimens cracked when exposed to deionized water which contained 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} .

Table 3.3.1.5.2

Stress Cracking of U-Bends in Deionized Water

Number of double U-bends tested	18
Number of U-bends which cracked	10

Conditions which caused cracking	<u>No. Cracked</u>	<u>No. Tested</u>
Presence of heat treatment scale . . .	2	6
Presence of 0.12 wt% Fe^{+3} + 0.065 wt% Ni^{+2}	2	6
Presence of heat treatment scale and 0.12 wt% Fe^{+3} + 0.065 wt% Ni^{+2} . . .	6	6

(All cracks were intergranular)

Conditions (static):

Temperature: 250°F
 Time: 112 hrs
 Solvent: initially air-saturated (no replenishment)
 Specimens: sensitized at 1200°F for 50 hrs, then U-bent.

Cracking of sensitized 304 stainless steel specimens appeared to be more severe in deionized water than in Dow solvent NS-1 under the same test conditions. Cracks propagated further into coupons exposed to D.I. water compared to those exposed to NS-1 solvent. Additionally, cracks propagated completely through some 1/8" $\times$ 3/4" coupons exposed to D.I. water which contained Fe^{+3} and Ni^{+2} . Although cracking which occurred on 304 stainless alloys exposed to Dow solvent NS-1 usually was visible to the naked eye, this cracking was observed more easily under a microscope at 10-100X magnification.

In summary, these tests in which time and temperature and metallurgical parameters were constant showed that Dow solvent NS-1 had no more adverse IGSSC effects than deionized water on sensitized 304 stainless steel.

Tests also were conducted at 275°F and for 300 hours to determine if stress corrosion of stainless steel would occur at temperatures 25°F higher and times 200 hours longer than those planned for the actual decontamination of Dresden-1. Both sensitized (1200°F, 50 hours) and unsensitized specimens were tested. Heat treated alloys were descaled for 12 hours in refluxing NS-1 solvent prior to being formed into U-bend specimens. Most stress corrosion data were measured on coupons 1/8" or less in thickness, although 5/8"-thick welded, sensitized 304 stainless steel also was tested. Tests were carried out with air-saturated NS-1 solvent. The solvent was not replenished with air after the test containers were sealed.

Following the test, specimens were examined for stress corrosion, and a representative number were evaluated metallographically as described in Appendix II. Stress corrosion cracking was not detected on any of the U-bend specimens tested; sensitized 304 and 304L stainless steel performed as well as the unsensitized alloys, as shown in Table 3.3.1.5.3.

Table 3.3.1.5.3

Stress Corrosion Data for U-Bends Exposed to
Dow Solvent NS-1 for 300 Hours at 275°F

<u>Two Stage Single U-Bends of</u>	<u>Number Tested</u>	<u>Results*</u>
304	14	No cracking
304 sens. and descaled	22	No cracking
304 L	17	No cracking
316	5	No cracking

Conditions (Static):

Temperature: 275°F

Time: 300 hours

Solvent: initially air-saturated (no replenishment)

Specimens: sensitized at 1200°F for 100 hrs, cooled
in furnace.

* Stereomicroscope and metallographic examinations
were used to confirm the results.

Seventy-three specimens from the 300 series alloys were tested at times and temperatures considerably in excess of those planned for the actual cleaning of Dresden-1, without incidence of stress corrosion.

The 5/8"-thick welded specimens were prepared by welding 304 stainless steel under high heat input and restraint to provide a stressed, sensitized specimen. Two surfaces on each specimen were ground to introduce the effects of cold-work. Examination of these specimens after they were exposed to NS-1 solvent showed no detectable crack initiation around the weld or in the 10-30 mil cold-worked surface layer. The specimens were inspected visually, and then were subjected to a liquid dye penetration test. Figure 3.3.1.5 shows a photograph of the specimens after they were treated with dye and sprayed with developer. No evidence of porosity in the coupons was apparent.

In summary, data obtained for U-bend and welded specimens show that stress corrosion is not expected to occur on 300 series stainless steel alloys during the chemical cleaning of the Dresden-1 primary reactor with Dow solvent NS-1. Conditions sufficiently severe to produce stress corrosion cracking on sensitized 304 stainless steel exposed to NS-1 solvent also would produce failure of the alloy in deionized water. Typical photomicrographs of the type of failures discussed in this section of the report may be found in Appendix I.

3.3.1.6 Wedge Open Loaded Tests

Tests were carried out to determine if pre-existing cracks under stress would propagate in Dresden-1 either during or after the primary reactor was cleaned with Dow solvent NS-1. Eight 304 stainless steel specimens were fabricated based on a wedge open loaded (WOL) fracture design which incorporated a fatigue crack under stress (Figure 3.3.1.6.1). Each specimen was sensitized 50 hours at 1200°F, and then was pre-cracked in fatigue according to ASTM method E399. Subsequently, specimens were loaded to stress intensities (K_I) of approximately 10, 25, and 32 Ksi root-inches. Compliance curves indicated the actual stress intensity factors were close to the desired values.

Two-stage testing of four of these specimens was carried out. Specimens initially were exposed to Dow solvent NS-1 at the planned cleaning temperature of 250°F for 225 hours (more than twice the planned cleaning time of 100 hours). The specimens were transferred unrinsed to a rocking titanium autoclave and exposed to deionized water (~0.3 ppm oxygen) for 300 hours at 500°F. The remaining four specimens served as controls, and were

Figure 3.3.1.5

5/8"-thick abraded 304 SS specimens
after they were subjected to liquid dye
penetrant (Developer present on surface
of specimens).

Figure 3.3.1.6.1
Schematic of a WOL Fracture Mechanics Specimen

NOTES:

1. "A" surfaces perpendicular and parallel as applicable within 0.003 TIR
2. The points of intersection of the crack-starter tip (cut 0.03" deep with jeweler's saw) shall intersect with the two specimen faces equally distant from loading and within 0.008".

tested in D.I. water under the same conditions used for the initial testing of the first four specimens in NS-1 solvent. Coupons stressed at K_I of 25 Ksi root-inches also were exposed to NS-1 solvent and deionized water to which ferric and nickelous ions had been added.

The results of the WOL tests are summarized in Table 3.3.1.6. Only the loads necessary to yield initial K_I values were tabulated, since on specimens where cracking was apparent the stress crack propagated perpendicularly to the plane of the initial pre-crack. The final loads on each specimen were determined in a tensile tester but the final K_I values were not calculated since no new crack growth had occurred. Relaxation of the initial K_I 's of 25 and 32 Ksi root-inches to lower final loads occurred as expected, because of the ductile nature of the 304 stainless steel specimens.

Visual examination indicated that crevice and pitting corrosion had occurred on one specimen stressed at 25 Ksi root-inches and exposed to deionized water that contained 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} . All other specimens remained shiny and unattacked over their outer surfaces.

Table 3.3.1.6

Stress Corrosion Cracking of WOL Specimens

Spec. No.	Test Environment	Initial Load, lb	Estimated K, ksi/ $\sqrt{\text{in}}$ ^a	Fatigue Crack Length a, in ^b	Initial K, ksi/ $\sqrt{\text{in}}$ ^c	Final Load, lb ^b	Results
1	Deionized H ₂ O	787	10.0	0.928	11.5	714	Stress cracks
2	Dow Solvent NS-1	787	10.0	0.893	10.6	692	No effect
3	Deionized H ₂ O	1970	25.0	0.880	25.4	1210	Crevice attack
4	Dow Solvent NS-1	1970	25.0 -	0.897	26.5	1475	No effect
5	Deionized H ₂ O 0.12% Fe ⁺³ 0.065% Ni ⁺²	1970	25.0	0.881	25.4	1190	Pitting, stress cracks
6	Dow Solvent NS-1 0.12% Fe ⁺³ 0.065% Ni ⁺²	1970	25.0	0.881	25.4	1280	Crevice attack
7	Deionized H ₂ O	2500	32.0	0.890	32.9	1320	Crevice attack
8	Dow Solvent NS-1	2500	32.0	0.881	32.2	1625	No effect

^aBased on nominal crack length of 0.84 in.

Note: Where cracks occurred they were IGSSC

^bMeasured after SCC test.

$$K = \frac{P}{BW^{1/2}} \left[29.6 \frac{d}{W^{1/2}} - 185.5 \frac{d^3}{W^{3/2}} + 655.7 \frac{d^5}{W^{5/2}} - 1017 \frac{d^7}{W^{7/2}} + 638.9 \frac{d^9}{W^{9/2}} \right]$$

where: P = Load in lbs

B = Specimen thickness in inches

W = Distance from loading line to back of specimen, in inches

d = Distance from loading line to crack tip, in inches

After each specimen was examined visually it was pulled apart and the stressed fatigue crack area was examined. Figure 3.3.1.6.2 defines the five regions of each specimen that are displayed in subsequent photographs (Figures 3.3.1.6.3 - 3.3.1.6.6). The region of most interest is region 2 where ductile tear resulted from bending the specimen open so that it could be cut apart without disturbing the precrack; this region indicates the boundary of the precrack. Also of interest is region 3, the area of fatigue precrack.

Photograph (a) in Figure 3.3.1.6.3 shows that stress corrosion propagated perpendicularly to the face of the fatigue crack in a specimen stressed at K_I of 11.5 Ksi root-inches, after that specimen was exposed only to deionized water. Photograph (b), however, shows that a similarly stressed specimen exhibited neither general attack nor corrosion on the fatigue crack surface when exposed to Dow solvent NS-1.

3.3.2 Type 400 Series Stainless Steels

Types 405, 410, 416, and 442 stainless steels of the 400 series alloys are present in Dresden-1. The corrosion performance of these alloys in Dow solvent NS-1, as well as the performance of 430 and 446 alloys, was evaluated in order to obtain as much information as possible about their behavior under the proposed cleaning conditions.

The 410 and 416 stainless steels are martensitic. They can be hardened by a heat treatment at 1850°F then followed by cooling in air or oil.

The other 400 series steels listed above are ferritic and are not subject to hardening by heat treatment near 1850°F followed by cooling in air or oil. The only heat treatment applied to truly ferritic steels in an annealing process used to relieve welding or cold working stress. Also, ferritic steels are subject to embrittlement when heated at 885°F for extended time periods.

3.3.2.1 Type 405 Stainless Steel

Evaluation of corrosion resistance properties of 405 stainless steel in Dow solvent NS-1 at times, temperature, and stress levels exceeding those anticipated in the decontamination of Dresden-1 showed no localized corrosion mechanisms or general corrosion rates of significant magnitude.

Figure 3.3.1.6.4 Photograph (a) shows that 304 stainless steel specimens stressed at K_I of ~25 Ksi root-inches and exposed to deionized water underwent crevice attack in the stressed region adjacent to the crack tip but did not exhibit stress corrosion. A similar specimen, shown in Photograph (b), exhibited no evidence of crevice or stress corrosion when exposed to NS-1 solvent.

Additional 304 stainless steel specimens stressed at K_I of ~25 Ksi root-inches were exposed to Deionized water and to Dow solvent NS-1 each of which contained 0.12 wt% Fe^{+3} and 0.065 wt% Ni^{+2} (Figure 3.3.1.6.5, Photographs (a) and (b), respectively). The specimen exposed to Deionized water which contained added impurity ions exhibited severe pitting over its entire surface, and showed evidence of crevice corrosion at the fatigue crack and extending perpendicularly into the ductile region. In addition, stress corrosion cracks occurred in region 4 where the specimen was cut to initiate the precrack. The specimen exposed to Dow solvent NS-1 at the same impurity level showed some evidence of corrosion. However the precrack did not extend the complete width of this specimen, and crevice attack actually did not occur. The retention of the original machining marks also indicates the passive nature of 304 stainless steel in Dow solvent NS-1.

A specimen of 304 stainless steel which had been stressed at K_I of 32.9 Ksi root-inches and exposed to deionized water exhibited crevice corrosion ahead of the precrack and accelerated attack on the face of the precrack, as shown in Figure 3.3.1.6.6, Photograph (a). Photograph (b) shows that a similarly stressed coupon exposed to Dow solvent NS-1 did not undergo crevice attack or attack on the precrack surface and retained a well-defined precrack boundary.

In summary, these tests indicated that Dow solvent NS-1 neither corroded stressed crevices of sensitized 304 stainless steel nor initiated stress corrosion from crevices of this alloy under conditions severe enough to cause stress initiation, pitting, and crevice corrosion on specimens exposed to deionized water. Individual corrosion data sheets are given in Appendix II.

Exposure of 405 stainless steel, as weight loss and single U-bend specimens, to Dow solvent NS-1 produced low corrosion rates as shown in Tables 3.3.2.1.1 and 3.3.2.1.2. Corrosion rates of 7 mpy and 2.9 mpy were obtained in 16 and 100 hour tests, respectively, at 250°F in the liquid phase. A 16 hour vapor phase test at 250°F gave a corrosion rate of 0.3 mpy. Five single U-bend specimens tested at 275°F for 100 hours exhibited average corrosion rates

Figure 3.3.1.6.2

Five regions are displayed in photographs of WOL specimens

Region 1: Area where the specimen was cut apart.

Region 2: Area of ductile tear which resulted from bending the specimen open so that it could be cut apart without disturbing the pre-crack.

Region 3: Area of fatigued pre-crack.

Region 4: Key-way cut to initiate fatigue pre-crack.

Region 5: Machined portion of the specimen leading to key-way cut.

Regions 3 and 4 are useful in evaluating the stress and crevice corrosion behavior of this type of specimen. (2.5X magnification)

Figure 3.3.1.6.3

WOL Fracture Mechanics Specimen
Tested at Stress Intensity of
Approximately 10 Ksi root-inches

(a)

WOL Specimen Stressed to 11.5 Ksi root-inches and Exposed to Air-Saturated Dow solvent NS-1 for 225 hrs at 255°F, then to de-ionized water at 500°F for 300 hrs. (2.5X magnification)

(b)

WOL Specimen Stressed to 10.6 Ksi root-inches and Exposed to Air-Saturated Dow solvent NS-1 for 225 hrs at 255°F then, to deionized water at 500°F for 300 hrs (note the absence of stress or crevice attack). (2.5X magnification)

Figure 3.3.1.6.4

WOL Fracture Mechanics Specimen Tested at
Stress Intensity of Approximately 25 Ksi root-inches

(a)

WOL Specimen Stressed to 25.4 Ksi root-inches and Exposed to Air-Saturated Deionized Water for 225 hrs at 255°F, then to Deionized Water at 500°F for 300 hrs (Note mild crevice attack, 30-60 mils deep ahead of pre-crack). (2.5X magnification)

(b)

WOL Specimen Stressed to 26.5 Ksi root-inches and Exposed to Air-Saturated Dow Solvent NS-1 for 225 hrs at 255°F, then to Deionized Water at 500°F for 300 hrs (Note absence of adverse attack). (2.5X magnification)

Figure 3.3.1.6.5

WOL Fracture Mechanics Specimens Tested at
Stress Intensity of Approximately 25 Ksi root-inches
With Ferric and Nickelous Ions Added

(a)

WOL Specimen Stressed to 25.4 Ksi root-inches and Exposed to Air-Saturated Deionized Water with 0.12% Ferric Ion and 0.065% Nickelous Ion for 225 hrs at 255°F, then to Deionized Water Without Impurities at 500°F for 300 hrs (Note extensive pitting and stress attack with pits F10 mils deep). (2.5X magnification)

(b)

WOL Specimen Stressed to 25.4 Ksi root-inches and Exposed to Air-Saturated Dow Solvent NS-1 with 0.12% Ferric Ion and 0.065% Nickelous Ion for 225 hrs at 255°F, then to Deionized Water at 500°F for 300 hrs (Note absence of stress attack or of crevice attack on pre-cracked surface, original fatigue crack did not extend full width of specimen). (2.5X magnification)

Figure 3.3.1.6.6

WOL Fracture Mechanics Specimens Tested at
Stress Intensity of Approximately 32 Ksi root-inches

(a)

WOL Specimen Stressed to 32.9 Ksi root-inches and Exposed to Air-Saturated Deionized Water for 225 hrs at 255°F, then to Deionized Water at 500°F for 300 hrs (Note crevice attack, 30-60 mils deep ahead of pre-crack). (2.5X magnification)

(b)

WOL Specimen Stressed to 32.2 Ksi root-inches and Exposed to Air-Saturated Dow Solvent NS-1 for 225 hrs at 255°F and then to Deionized Water at 500°F for 300 hrs (Note absence of adverse attack). (2.5X magnification)

Table 3.3.2.1.1

Weight Loss Corrosion Rates of 400 Series SS
in Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>100-Hour Corrosion Rate (mpy)</u>
405 SS	7.0, 7.0	2.9
410 SS	5.5, 6.4	
416 SS	9.9 ^a , 12 ^b , 13 ^b , 13 ^a , 9.8 ^a	6.1 ^a
442 SS	0.36, 0.41	

^aMinor pitting

^bExtensive pitting, with some pits as large as 30 mils in diameter and 14 mils in depth.

Table 3.3.2.1.2

General Corrosion Rates of 400 Series SS
in Dow Solvent Liquid and Vapor NS-1 at 250°F

<u>Alloy</u>	<u>Phase</u>	<u>16-Hour Corrosion Rate (mpy)</u>
405 SS	Vapor	0.33
405 SS	Liquid	6.7
410 SS	Vapor	0.08
410 SS	Liquid	8.7

of 3.2 mpy, as shown in Table 3.3.2.1.3. Type 405 stainless steel embrittled at 885°F corroded at an average rate of 6.3 mpy in tests of five single U-bend specimens under the same conditions.

No significant crevice corrosion was found on 405 stainless steel specimens which included metal to metal crevices, TEFLON to metal crevices, or rubber to metal crevices. Corrosion rates for a TEFLON to metal crevice are given in Table 3.3.2.1.4.

Galvanic couples of 405 stainless steel bolted or rubber-banded to several other metals were tested as shown in Tables 3.3.2.1.5 through 3.3.2.1.10. The alloy was consistently anodic toward carbon steels. Type 1020 carbon steel in 1:1 area couples with 405 stainless steel corroded at 21-45 mpy when exposed to Dow solvent NS-1 at 250°F; the 405 stainless alloy corroded at 170-354 mpy. At an increased area ratio of 10:1 in favor of 1020 carbon steel, the couples showed corrosion rates of 12 mpy for the carbon steel and 877 mpy for the 405 stainless alloy.

Couples of 405 stainless steel with ASTM SA 36 structural steel, at a 1:1 area ratio, gave 100 hour corrosion rates of 355-392 mpy and 137-159 mpy, respectively, in NS-1 solvent. Couples of the 405 alloy with ASTM SA 516 Grade 70 pressure vessel plate had 100 hour corrosion rates of 263-321 mpy and 196-200 mpy, respectively. No explanation is apparent for this consistently anodic behavior of 405 stainless steel toward carbon steel, but the corrosion rates do not appear to be significant enough to warrant concern for the proposed cleaning.

Ferric ion added to Dow solvent NS-1 had the effect of increasing the corrosion rate of 405 stainless steel alone. However, in a 1:1 galvanic couple of 405 alloy with 1020 carbon steel, the corrosion rate of the 1020 steel increased with ferric ion concentration, and the corrosion rate of the 405 alloy decreased, until at 0.5% ferric ion the 1020 steel became anodic to the 405 alloy (Table 3.3.2.1.6).

Galvanic couples of 405 stainless steel to other 400 alloys and to 304L stainless steel resulted in corrosion rates of 15 mpy or less, as shown in Table 3.3.2.1.8.

Double U-bend specimens exposed to simulated used NS-1 solvent for 300 hours at 250°F gave no indication of stress cracking, as shown in Table 3.3.2.1.9.

Table 3.3.2.1.3

Single U-Bend Corrosion Data on Type
400 Series Stainless Steels Exposed
to Dow Solvent NS-1 at 275°F

<u>Alloy and Heat Treatment</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Stress Results</u>
405 - none	3.6	No cracking
405 - none	2.6	No cracking
405 - none	2.5	No cracking
405 - none	3.0	No cracking
405 - none	4.2	No cracking
405 - 885°F - 100 hours ^a	4.4	No cracking
405 - 885°F - 100 hours	7.4	No cracking
405 - 885°F - 100 hours	6.0	No cracking
405 - 885°F - 100 hours	7.2	No cracking
405 - 885°F - 100 hours	6.6	No cracking
410 - none	4.3	No cracking
410 - none	4.6	No cracking
410 - none	4.1	No cracking
410 - none	3.8	No cracking
410 - none	6.4	No cracking
416 - none	398	No cracking (stress etching)
416 - none	415	No cracking (stress etching)
416 - none	439	No cracking (stress etching)
416 - none	265	No cracking (stress etching)
416 - none	569	No cracking (stress etching)

^aTemper embrittlement; descaled in Dow solvent NS-1 before
U-bends were formed.

Table 3.3.2.1.3 (Continued)

<u>Alloy and Heat Treatment</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Stress Results</u>
430 - none	0.91	No cracking
430 - none	1.3	"
430 - none	0.81	"
430 - none	1.0	"
430 - none	1.3	"
430 - 885°F - 100 hours ^a	2.8	"
430 - 885°F - 100 hours	2.1	"
" " " "	2.0	"
" " " "	2.3	"
" " " "	1.8	"
442 - 885°F - 100 hours ^a	1.4	"
442 - 885°F - 100 hours	1.8	"
" " " "	1.7	"
" " " "	1.0	"
" " " "	1.5	"
446 - none	0.25	"
" "	0.23	"
" "	0.09	"
" "	0.33	"
" "	0.53	"
446 - 885°F - 100 hours ^a	1.5	"
446 - 885°F - 100 hours	3.0	"
" " " "	3.0	"

^aTemper embrittlement; descaled in Dow solvent NS-1 before U-bends were formed.

Table 3.3.2.1.4

General Corrosion Rates of 400 Series SS
With Artificial Crevice, in Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>Results</u>	<u>100-Hour Corr. Rate (mpy)</u>	<u>Results</u>
405 SS	6.1, 5.2	No crevice attack		
416 SS	21, 10	Pits and crevice attack	5.1	Minor pitting, no crevice attack

Table 3.3.2.1.5

General Corrosion Rates for Galvanic
Couples of 1020 Steel and 400 Series SS
in Dow Solvent NS-1 at 250°F

Galvanic Couple ^a 1020:400 Series SS		16-Hour CR (mpy)	100-Hour CR (mpy)
1020)	Rubber-banded together	54, 61	45, 21
405 SS)		248, 294	354, 170
1020)	Bolted together with TEFLON spacer	58	
405 SS)		262	
1020)	Rubber-banded together 1020:405 SS ~10:1 area	11, 52	12
405 SS)		283, 445	877
1020)	Rubber-banded together	89, 55, 128, 57	46, 41, 48, 28
410 SS)		150	
		79, 233, 23, 103, 23	370, 325, 99, 115
1020)	Bolted together with TEFLON spacer	47	95
410 SS)		181	395
1020)	Rubber-banded together 1020:410 SS ~10:1 area ratio	41	
410 SS)		427	
1020)	Rubber-banded together	84, 62	22
416 SS)		135, 153	157
1020)	Bolted together with TEFLON spacer	62	
416 SS)		308	
1020)	Rubber-banded together	112, 120	
442 SS)		0.57, 1.0	

^a~1:1 area ratio unless stated otherwise.

Table 3.3.2.1.6

Effect of Ferric Ion on General Corrosion Rates
 of 405 SS Alone and Galvanically Coupled to 1020
 Carbon Steel, in Dow Solvent NS-1 at 250°F

Alloy	16-Hour Corrosion Rate (mpy)		
	0%	0.1%	0.5%
	<u>Ferric Ion</u>	<u>Ferric Ion</u>	<u>Ferric Ion</u>
405 SS	7.0 (avg)	29	45
1020) Rubber-banded together	58 (avg)	110	358
405 SS) (1:1 area ratio)	268 (avg)	225	128

Table 3.3.2.1.7

General Corrosion Rates for Galvanic
Couples of Carbon Steels and 400 Series SS
at 250°F for 100 hrs in Dow Solvent NS-1

<u>Galvanic Couple (1:1 Area Ratio)</u>	<u>Corrosion Rate (mpy)</u>
ASTM SA 36) 405 SS) Rubber-banded together	159 355
ASTM SA 36) 405 SS) Bolted together	137 392
ASTM SA 516 Gr70) 405 SS) Rubber-banded together	200 263
ASTM SA 516 Gr70) 405 SS) Bolted together	196 321
ASTM SA 516 Gr70) 410 SS) Rubber-banded together	205 335
ASTM SA 516 Gr70) 410 SS) Bolted together	218 334
405 SS	2.7
410 SS	2.3

Table 3.3.2.1.8

General Corrosion Rates for 400 Series SS
Galvanically Coupled to itself and to 304L SS,
in Dow Solvent NS-1 at 250°F

Galvanic Couple	16-Hour Corrosion Rate (mpy)
405 SS) 410 SS) Rubber-banded together	9.2 10
405 SS) 416 SS) Rubber-banded together	16 35
405 SS) 405 SS) Rubber-banded together	14 15
405 SS)	14
405 SS) 304L SS) Rubber-banded together	2.3 2.1 0.36 0.84
410 SS) 304L SS) Rubber-banded together	3.3 0.31

Table 3.3.2.1.9

Double U-Bend Stress Corrosion Data on Type 400
Series Stainless Steels at 250°F in Dow Solvent
NS-1 With and Without Ferric and Nickelous Ions Added

<u>Specimen</u>	<u>Test Solution</u>	<u>Time, Hours</u>	<u>Results</u>
405 SS	Simulated used Dow Solvent NS-1 ^a	300	No cracking
410 SS	Dow Solvent NS-1	140	No cracking
416 SS	Dow Solvent NS-1	140	No cracking, etching along stress bands
446 SS	Simulated used Dow Solvent NS-1	300	No cracking

^aDow solvent NS-1 plus 0.12 wt% Fe⁺³ as ferric ammonium sulfate
and 0.065 wt% Ni⁺² as nickel sulfate.

Table 3.3.2.1.10

Galvanic Corrosion Data on Stressed Double U-Bends
of 400 Series SS and 304 SS in Dow Solvent NS-1
Plus 0.12 wt% Ferric Ion and 0.065 wt% Nickelous
Ion at 250°F for 100 hrs, Then in Deionized,
Deoxygenated Water at 500°F for 200 hrs.

<u>Galvanic Double U-Bend</u>	<u>CR (mpy) of Alloy Coupled to 304 SS</u>	<u>Stress Results</u>
405 SS inner (1:5 area ratio) 304 SS outer	3.3, 3.1, 3.1, 3.7	No cracking
410 SS inner (1:5 area ratio) 304 SS outer	5.7, 4.9, 5.3, 5.5	No cracking
446 SS inner (1:5 area ratio) 304 SS outer	0.1, 0.1, 0.1, 0.1	No cracking

Test media simulated used Dow Solvent NS-1.

U-bend galvanic couples were made by placing a 3/4" x 3" specimen of 400 series alloy inside a 2" x 6" 304 SS specimen and simultaneously bending the specimens. The resulting curvature plus the bolting together of the outer 304 SS specimen was sufficient to hold the inner specimen in place.

Galvanic and stress corrosion data are given in Table 3.3.2.1.10 for a 405 stainless steel inner U-bend coupled to a 304 stainless steel outer U-bend at an area ratio of 1:5, respectively. When these specimens were exposed to simulated used Dow solvent NS-1 for 100 hours at 250°F and then to deionized, deoxygenated water for 200 hours at 500°F, the 405 alloy corroded at slightly >3 mpy with no indication of stress cracking.

Type 405 stainless steel specimens, both annealed and heat treated at 885°F to produce embrittlement, showed no stress corrosion cracking tendencies when tested in Dow Solvent NS-1 for 100 hours at 275°F (Table 3.3.2.1.3). Metallographs of these specimens are shown in Figures 3.3.2.1.1 and 3.3.2.1.2. Figure 3.3.2.1.3 shows a metallograph of 405 stainless steel coupled in a double U-bend with 304 stainless steel and exposed to simulated used NS-1 solvent at 250°F followed by exposure to deionized, deoxygenated water at 500°F. No indication of serious attack or stress cracking was found.

In summary, 405 stainless steel did not exhibit significant weight loss or crevice, galvanic, or stress corrosion in Dow solvent NS-1 under conditions expected to occur during the decontamination of Dresden-1. Although 405 stainless steel was consistently anodic toward carbon steel in the proposed cleaning environment, this behavior is not expected to be a problem during the decontamination.

3.3.2.2 Type 410 Stainless Steel

As a result of the test program, no adverse corrosion effects are expected for 410 stainless steel exposed to Dow solvent NS-1 at the times and temperatures projected for the Dresden-1 decontamination. Individual corrosion data sheets are given in Appendix I.

General corrosion rates of 410 stainless steel were approximately the same (4-8 mpy) as those observed for 405 stainless steel (Tables 3.3.2.1.1, 3.3.2.1.2, and 3.3.2.1.3). No crevice corrosion under TEFLON washers was noted in U-bend tests or in metal crevices in galvanic tests where specimens were bolted together with a TEFLON spacer or which were rubber-banded together.

Figure 3.3.2.1.1

400X metallograph of type 405 SS exposed to Dow solvent NS-1 for 100 hours at 275°F. Metallograph taken from area of greatest stress of single U-bend specimen. Metal structure shows no serious attack nor any indication of stress cracking.

XXIX-8

Electrolytic
HCl Methanol Etch

Figure 3.3.2.1.2

400X metallograph of 885°F embrittled type 405 SS exposed to Dow solvent NS-1 for 100 hours at 275°F. Metallograph taken from area of greatest stress of single U-bend specimen. Metal structure shows no serious attack nor any indication of stress cracking.

XXX-5

Vilello's Reagent Etch

Figure 3.3.2.1.3

400X metallograph of inner U-bend specimen of 405 SS coupled to outer U-bend specimen of 304 SS and exposed to Dow solvent NS-1 plus 0.12% ferric ion and 0.065% nickel ion at 250°F for 100 hours, and then exposed to deionized, deoxygenated water at 500°F for 200 hours. Metallograph taken from Area of greatest stress of single U-bend specimen. Metal structure shows no sign of serious attack nor any indication of stress cracking.

LIV-

Etch

Galvanic behavior of 410 stainless steel coupled to 1020 carbon steel was erratic in 16 hour tests with Dow solvent NS-1, but the alloy consistently was anodic to all carbon steels in 100 hour tests. (Tables 3.3.2.1.5 and 3.3.2.1.7). When coupled to 405 stainless steel, the 410 alloy corroded at 10 mpy in 16 hour tests at 250°F with NS-1 solvent, as shown in Table 3.3.2.1.8. Low corrosion rates were exhibited by the 410 alloy in double U-bend couples with 304 stainless steel, as shown in Table 3.3.2.1.10.

Double U-bends of 410 stainless steel exposed to Dow solvent NS-1 for 140 hours at 250°F showed no indication of stress cracking (Table 3.3.2.1.9). In combination galvanic and stress tests, 410 steel inner U-bend and 304 steel outer U-bend specimens exposed to simulated used Dow solvent NS-1 for 100 hours at 250°F and then to deoxygenated, deionized water at 500°F for 200 hours showed no indication of stress cracking (Table 3.3.2.1.10). Single U-bends of 410 steel exposed to Dow solvent NS-1 for 100 hours at 275°F gave low weight loss rates and showed no indication of stress cracking (Table 3.3.2.1.3).

3.3.2.3 Type 416 Stainless Steel

General corrosion rates for weight loss specimens of 416 stainless steel were only slighter higher than those of 405 or 410 alloys, although some pitting was observed (Table 3.3.2.1). Weight loss rates of stressed single U-bend specimens at 275°F were significantly higher, e.g., 265-569 mpy; Stress etching occurred on the outer surfaces of the U-bends.

Light crevice attack was observed on some specimens which contained TEFLON spacers (Table 3.3.2.1.4). When galvanically coupled to 1020 carbon steel at a 1:1 area ratio, 416 stainless steel was consistently the anodic member of the couple (Table 3.3.2.1.5). Corrosion rates in NS-1 solvent varied from 135-308 mpy for the 416 alloy and from 22-84 mpy for the 1020 steel. Additionally, 416 stainless steel was slightly anodic when coupled to 405 stainless steel at a 1:1 area ratio (Table 3.3.2.1.8).

Single U-bends of 416 stainless steel exposed to Dow solvent NS-1 at 250°F for 140 hours and at 275°F for 100 hours did not undergo stress cracking, although etching was noted along the stress bands on the outer surfaces of the U-bends (Tables 3.3.2.1.3 and 3.3.2.1.9).

Type 416 stainless steel corroded more in Dow solvent NS-1 than other 400 series alloys tested. This martensitic stainless steel contains sulfur which improves the machinability of the alloy but which also probably contributed to the accelerated attack of the stressed specimen. However these corrosion rates are not high enough to cause concern for the decontamination process at the projected times and temperatures. Individual corrosion data sheets are contained in Appendix II.

3.3.2.4 Type 430 Stainless Steel

The higher chromium content of ferritic 430 stainless steel, compared to some other 400 series alloys, contributed to the low general corrosion rates found for U-bend specimens of the alloy exposed to Dow solvent NS-1 for 100 hours at 275°F (Table 3.3.2.1.3). General corrosion rates averaged 1.1 mpy for annealed specimens and 2.2 mpy for specimens embrittled at 885°F. Neither type of specimen underwent detectable stress cracking. Individual corrosion data sheets are given in Appendix I.

3.3.2.5 Type 442 Stainless Steel

Type 442 stainless steel, a heat resistant ferritic alloy, had general corrosion rates of 1 mpy or less when tested alone or galvanically coupled to 1020 carbon steel (Tables 3.3.2.1.1 and 3.3.2.1.5). Single U-bend specimens embrittled at 885°F had average weight losses of 1.5 mpy (Table 3.3.2.1.3).

No crevice attack by NS-1 solvent was noted on specimens galvanically coupled to 1020 steel or underneath TEFLON washers which insulated the bolts used to apply stress to the single U-bend specimens. Temper-embrittled single U-bend specimens showed no indication of stress cracking in Dow solvent NS-1 at 275°F (Table 3.3.2.1.3). This alloy is not expected to corrode significantly in Dow solvent NS-1 at the times and temperatures projected for Dresden-1 decontamination. Individual corrosion data sheets are given in Appendix II.

3.3.2.6 Type 446 Stainless Steel

Type 446 alloy is a heat resistant ferritic stainless steel which has a higher chromium content than 442 stainless steel. General corrosion rates averaged 0.3 mpy for single U-bends exposed to Dow solvent NS-1 for 100 hours at 275°F; stress cracking did not occur. Specimens embrittled at 885°F averaged 2.5 mpy under the same condi-

tions, as shown in Table 3.3.2.1.3. Embrittled specimens which did not crack when formed into U-bends did not stress crack in NS-1 solvent at 275°F.

Double U-bends in which 446 stainless steel (inner U-bend) was coupled to 304 stainless steel (outer U-bend) corroded at rates of 0.1 mpy when exposed 100 hours to simulated used Dow solvent NS-1 at 250°F and then for 200 hours to deionized, deoxygenated water at 500°F (Table 3.3.2.1.10). The specimens showed no indication of stress cracking under these conditions nor when exposed to simulated used NS-1 solvent for 300 hours at 250°F (Table 3.3.2.1.9).

Based on these results, 446 stainless steel alloy is not expected to corrode significantly when exposed to Dow solvent NS-1 at times and temperatures projected for decontamination of Dresden-1. Individual corrosion data sheets are given in Appendix I.

3.3.3 All Other Materials

Corrosion results for all other materials tested in Dow solvent NS-1 are given in this section. Many of these construction materials are present only in small amounts in Dresden-1, and are less significant from a corrosion standpoint than are the 300 or 400 series stainless steels. However, a knowledge of the behavior of these other materials in Dow solvent NS-1 is important to ensure the success of the decontamination.

3.3.3.1 Carbon Steels (AISI 1020, AISI B-1213, ASTM SA 36 and ASTM SA 516 Grade 70).

Carbon steel is not a major construction material in Dresden-1 except where it occurs as the backing material for 304 stainless steel cladding. Only in imperfect areas of cladding will carbon steel come in contact with Dow solvent NS-1 during the decontamination. Most of the corrosion tests described here were performed with AISI 1020 carbon steel. A lesser amount of testing was carried out on AISI B-1213 (resulfurized carbon steel), ASTM SA 36 structural steel, and ASTM SA 516 Grade 70 pressure plate.

The initial sample of cleaning solvent (I_r solution, Table 3.3.3.1.1) was insufficiently inhibited, as shown in tests on AISI 1020 carbon steel, although this solution was not aggressive to 300 series stainless steels at 250°F. The choice of inhibitors was somewhat limited because of the low chloride requirement dictated by the 300 series stainless steels present in Dresden-1, but

Table 3.3.3.1.1

General Corrosion Rates of 1020 Carbon Steel
Exposed to First Proposed Decontamination
Solvent at 250°F

Test Solvent	Length of Test (Hours)	Corrosion Rate (mpy)
I_r solution	16	1093
I_r solution + Al45 inhibitor	16	36
I_r solution	72	467
I_r solution + Al45 inhibitor	72	25
I_r solution + Al75 inhibitor	100	177
I_r solution + Al45 inhibitor	100	24

Al45, a Dow-developed material, was found to be a suitable corrosion inhibitor for NS-1 solvent. As described in the section on 300 series stainless steels, the inhibitor concentration was increased from standard to 2x standard to ensure maximum protection against accidental temperature excursions up to approximately 25°F higher than the planned decontamination temperature.

General corrosion rates of 1020 carbon steel weight loss specimens in NS-1 solvent consistently were <50 mpy as shown in Tables 3.3.3.1.2 and 3.3.3.1.3. Specimens exposed to the vapor above NS-1 solvent, in the presence of air, corroded at somewhat higher rates. Corrosion rates decreased significantly when a nitrogen atmosphere was used in both liquid and vapor phase tests. Corrosion rates of B-1214 resulfurized steel, although significantly higher than those of 1020 carbon steel, still were acceptable and also were decreased by use of a nitrogen atmosphere.

Artificial crevice tests indicated little pitting or crevice attack occurred on 1020 or B-1213 alloys in Dow solvent NS-1, as shown in Table 3.3.3.1.4. Little indication of crevice attack also was observed on galvanic couples that contained the carbon steels described in Tables 3.3.3.1.5 through 3.3.3.1.7. Type 1020 steel was anodic toward 304 stainless steel, and in 1:1 area ratio couples corroded at a rate of 125-200 mpy compared to <50 mpy for the 1020 alloy alone (Table 3.3.3.1.5). Type 1020 carbon steel coupled at a 1:30 ratio with 304L stainless steel respectively, corroded at >1000 mpy. Severe attack of 1020 steel coupled galvanically to 304 stainless steel in Dresden-1 is not expected, however, since 1:30 ratios of the two alloys are unlikely to occur in the reactor. Similar corrosion rates were obtained in 16 and 100 hour exposures of specimens to NS-1 solvent. The presence of a nitrogen atmosphere did not decrease the corrosion rates of galvanic couples as it did for ordinary weight loss specimens.

Corrosion rates of 1020 carbon steel galvanically coupled to 400 series stainless steels were discussed in conjunction with Tables 3.3.3.1.5 and 3.3.3.1.7, where data showed that 405, 410, and 416 stainless steels were anodic to 1020 carbon steel in Dow solvent NS-1.

Data are shown in Table 3.3.3.1.6 for galvanic couples of other carbon steels to 304L stainless steel. The corrosion rates of SA 516 Grade 70 and SA 36 are somewhat higher than those of 1020 carbon steel, probably because of higher carbon contents. B-1213 also showed higher corrosion rates, possibly because of its sulfur content.

Table 3.3.3.1.2

General Corrosion Rates of Carbon Steels
Exposed to Dow Solvent NS-1 at 250°F

Alloy	16-Hour Corrosion Rate (mpy)	100-Hour Corrosion Rate (mpy)
AISI 1020	12, 9.6	14
AISI 1020 - N ₂ sat'd.	4.2	
AISI B-1213 (resulfurized carbon steel)	42, 50, 24, 68	102

Table 3.3.3.1.3

General Corrosion Rates of Carbon Steels
in Dow Solvent NS-1 Liquid and Vapor at 250°F

<u>Alloy</u>	<u>Phase</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>100-Hour Corrosion Rate (mpy)</u>
1020	Vapor	29, 51, 60, 11, 25	6.1
1020	Liquid	14, 15, 48, 5, 11	36
1020	Vapor - N ₂ sat'd.	2.1	
1020	Liquid - N ₂ sat'd.	5.5	
B-1213	Vapor	15	
B-1213	Liquid	55	
B-1213	Vapor - N ₂ sat'd.	1.8	
B-1213	Liquid - N ₂ sat'd.	7.9	

Table 3.3.3.1.4

General Corrosion Rates of Carbon Steels
With Artificial Crevice, in Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>Crevice Results</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Crevice Results</u>
1020	11, 12	No crevice attack		
B-1213	47, 30	No crevice attack	109, 105	Edge attack no crevice attack

Table 3.3.3.1.5

General Corrosion Rates for Galvanic Couples of 1020
Carbon Steel and 304L SS in Dow Solvent NS-1 at 250°F

Galvanic Couple ^a		16-Hour Corrosion Rate ^b (mpy)	100-Hour Corrosion Rate (mpy)
1020) 304L SS)	Welded ^b	138, 174 -	
1020) 304L SS)	Welded, N ₂ sat'd. ^b	127 -	
1020) 340L SS)	Rubber-banded together	188, 175, 166 1.3, 1.0, 1.4	139, 156, 87 5.1, 4.3, 0.94
1020) 304L SS)	Rubber-banded together, N ₂ sat'd.	187 3.0	
1020) 304L SS)	Bolted together with TEFLON spacer	216 1.3	
1020) 304L SS)	Bolted together with TEFLON spacer, N ₂ sat'd.	173 2.6	
1020) 304L SS)	Rubber-banded together 1020:304L SS ~10:1 area ratio	38, 43, 45 0.45, 25, 1.5	
1020) 304L SS)	Rubber-banded together 1020:304L SS ~1:10 area ratio	799, 726 0.71, 0.53	771 2.4
1020) 304L SS)	Rubber-banded together 1020:304L SS ~1:30 area ratio	1550 0.50	

^aArea ratio ~1:1 unless stated otherwise.

^bAll weight loss assigned to anodic member of welded couples.

Table 3.3.3.1.6

General Corrosion Rates for Galvanic Couples
of Carbon Steels and 300 Series SS Alloys in
Dow Solvent NS-1 at 250°F

<u>Galvanic Couple - Carbon Steel:300 Series SS</u>		<u>Area Ratio</u>	<u>100-Hour Corrosion Rate (mpy)</u>
SA 516 Gr 70) 304L SS)	Rubber-banded together	1:1	489 2.1
SA 516 Gr 70) 304L SS)	Bolted together	1:1	533 1.3
SA 516 Gr 70) 304L SS)	Rubber-banded together	1:10	1871 ^a 4.7 ^a
SA 516 Gr 70) Welded to itself) 304L SS)	Rubber-banded together	1:1	418, 400 3.9, 0.12
SA 516 Gr 70) Welded to itself) 304L SS)	Bolted together	1:1	493, 455, 478 2.5, 4.8, 5.2
SA 516 Gr 70) 304L SS, sensitized)	Rubber-banded together	1:1	489 1.5
SA 516 Gr 70) 304L SS, sensitized)	Bolted together	1:1	521 2.1
SA 36) 340L SS)	Rubber-banded together	1:1	453 3.5
SA 36) 304L SS)	Bolted together	1:1	511 2.4
SA 36) Welded to itself) 304L SS)	Rubber-banded together	1:1	493 16
SA 36) Welded to itself) 304L SS)	Bolted together	1:1	500, 467, 411 5.0, 6.1, 5.6

^aAverage of 8 test couples

Table 3.3.3.1.6 (Continued)

<u>Galvanic Couple - Carbon Steel:300 Series SS</u>		<u>Area Ratio</u>	<u>100-Hour Corrosion Rate (mpy)</u>
1020) 304L SS)	Rubber-banded together	1:1	204 2.0
1020) 304L SS)	Bolted together	1:1	63 1.4
1020) 304L SS)	Welded together	1:1	120 ^b -
1010 welded) to itself) 304L SS)	Rubber-banded together	1:1	166, 212, 291 8.5, 7.1, 5.8
1010 welded) to itself) 340L SS)	Bolted together	1:1	424, 263 9.0, 8.2
B-1213) 304L SS)	Rubber-banded together	1:1	398 3.4
D-1213) 304L SS)	Bolted together	1:1	1249 7.5

^bTotal weight loss assigned to 1020 portion of welded specimen.

Table 3.3.3.1.7

General Corrosion Rates for Galvanic Couples of 1020
Steel and Various Alloys in Dow Solvent NS-1 at 250°F

Galvanic Couple (1:1 Area Ratio)	16-Hour Corrosion Rate (mpy)
1020) 304 SS) Rubber-banded together	159, 164 0.8, 1.2
1020) 316 SS) Rubber-banded together	160 0.3
1020) 347 SS) Rubber-banded together	194 0.4
1020) INCONEL ^a 600) Rubber-banded together	70, 48 0.16, 0.28
1020) INCONEL 600) Bolted together with TEFLON spacer between	70 6.1
1020) INCOLOY 800) Rubber-banded together	115, 81 0.21, 0.84
1020) INCOLOY 800) Bolted together with TEFLON spacer between	139 1.4
1020) HASTELLOY ^b B) Rubber-banded together	119 0.00
1020) 17-4 P.H.) Rubber-banded together	141, 147 0.6, 0.4
1020) 70:30 Cu:Ni) Rubber-banded together	19 1.7
1020) Al 1100) Rubber-banded together	1.8 112
1020) Al 1100) Bolted together with TEFLON spacer between	2.9, 2.7 153, 163

^aTrademark of Huntington Alloys, Inc.

^bTrademark of Stellite Division, Cabot Corporation

No severe attack of these alloys is expected when Dresden-1 is decontaminated with Dow solvent NS-1. Corrosion rates for galvanic couples of 1020 carbon steel with other alloys are shown in Table 3.3.3.1.7. In general, the carbon steel performed similarly in couples with 304, 304L, and 315 stainless steels, and INCONEL 600, INCOLOY 800, and HASTELLOY B. Corrosion rates of the carbon steel in couples with Inconel 600 were somewhat lower than in couples with the other alloys. Additionally, 1020 steel did not corrode significantly in galvanic couples with copper-nickel 70-30. Aluminum 1100 provided galvanic protection to 1020 steel.

The presence of ferric ion increased the corrosion rate of 1020 carbon steel alone and coupled to 304L stainless steel when the specimens were exposed to Dow NS-1 solvent for 16 hours at 250°F (Table 3.3.3.1.8).

Single U-bends to 1020 steel exposed to NS-1 solvent at 275°F gave acceptable corrosion rates and did not exhibit stress cracking (Table 3.3.3.1.9 and Figure 3.3.3.1). Lower corrosion rates were achieved by doubling the standard inhibitor concentration in NS-1 solvent. Tests performed with stressed 304 and 304L stainless steel U-bends at 275°F also had shown that lower corrosion rates were obtained at double the inhibitor concentration compared to the originally recommended concentration.

Compared to the original specimens, annealed and quenched specimens of 1020 carbon steel gave acceptable although slightly higher corrosion rates, with no indication of stress cracking. Carbon steel B-1213, which has sulfur added to give improved machinability, also corroded at higher rates than the 1020 alloy. The added sulfur compounds usually are aligned in the direction in which the alloy is rolled, and produce a susceptibility to formation of elongated pits in the direction of rolling and round pits at right angles to the direction of rolling. Bending this alloy into a U-bend configuration disrupts the surface film and allows pitting or stress etching to occur. As discussed earlier, a nitrogen atmosphere reduced the overall rate of the specimens exposed to NS-1 solvent compared to specimens tested with air present.

Type 1020 carbon steel also was tested in Dow solvent NS-1 and simulated used solvent at two inhibitor levels and with the inhibitor omitted, as shown in Tables 3.3.3.1.10. This steel exhibited increased corrosion rates when the inhibitor was omitted, but neither the general attack or nor the stress corrosion cracking effects were unacceptable. The SA 516 Grade 70 specimens gave results as shown in Table 3.3.3.1.11.

Table 3.3.3.1.8

Effect of Ferric Ion on General Corrosion Rates of
1020 Carbon Steel alone and Galvanically coupled
to 304 ss, in Dow Solvent NS-1 at 250°F

Alloy	16-Hour Corrosion Rate (mpy)		
	0%	0.1%	0.5%
	<u>Ferric Ion</u>	<u>Ferric Ion</u>	<u>Ferric Ion</u>
1020	11 (avg.)	101	344
1020) Rubber banded together	179 (avg.)	231	464
304L SS) (1:1 area ratio)	1.7 (avg.)	0.7	0.8

Table 3.3.3.1.9

Single U-Bend Corrosion Data on Carbon
Steels Exposed to Dow Solvent NS-1 at 275°F

<u>Alloy and Heat treatment</u>	<u>Inhibitor Level</u>	<u>100-Hour Cor. Rate Stress (mpy)</u>	<u>Results</u>
1020 - none	"standard"	156	No cracking
1020 - none	"	240	No cracking
1020 - none	"	295	No cracking
1020 - none	"	236	No cracking
1020 - none	"	349	No cracking
1020 - 1650°F for 1 hr., water quench	"	255	No cracking
1020 - 1650°F for 1 hr., water quench	"	388	No cracking
1020 - 1650°F for 1 hr., water quench	"	443	No cracking
1020 - 1650°F for 1 hr., water quench	"	511	No cracking
1020 - 1650°F for 1 hr., water quench	"	416	No cracking
1020 - none	"2x standard"	105	No cracking
1020 - none	"	199	No cracking
1020 - none	"	121	No cracking
B-1213 - none	"	456	No cracking (stress etching)

Figure 3.3.3.1

400X Metallograph of AISI 1020 Carbon Steel exposed to Dow solvent NS-1 at 275°F for 100 hours.

Metallograph was taken from area of greatest stress on single U-bend specimen. Some surface etching is apparent but metal structure shows no serious attack nor any indication of stress cracking.

XXX-15

Picral Etch

Table 3.3.3.1.10

General Corrosion Rates and Stress Corrosion Results
on 1020 Carbon Steel in Dow Solvent NS-1 and
Simulated Used Dow Solvent NS-1 at 250°F
as a Function of Inhibitor Concentration

<u>Inhibitor Concentration</u>		<u>Contaminants</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Single U-Bend Stress Results</u>
0.0		None	510, 497	No cracking
standard ^a		None	31, 23	No cracking
2x standard ^b		None	27, 16	No cracking
0.0	0.12% Fe ⁺ ₃ , 0.065% Ni ⁺ ₂ (c)		602, 569	No cracking
standard ^a	"	"	244, 228	No cracking
2x standard ^b	"	"	50, 62	No cracking

^aoriginal recommended use concentration

^bconcentration recommended as a result of corrosion studies described
in this report

^csimulated used Dow solvent NS-1

Table 3.3.3.1.11

General Corrosion Rates on ASTM SA 516 Grade 70
Pressure Vessel Plate in Dow Solvent NS-1
and Simulated Used Dow Solvent NS-1
at 250°F as a Function of
Inhibitor Concentration

Inhibitor Concentration	Contaminants	100-Hour Corrosion Rate (mpy)
0.0	None	614, 500
standard ^a	None	423, 410
2x standard ^b	None	386, 387
0.0	0.12% Fe ⁺³ , 0.065% Ni ⁺² (c)	609, 623
standard ^a	" "	498, 566
2x standard ^b	" "	591, 504

^aoriginal recommended use concentration

^bconcentration recommended as a result of corrosion studies
described in this report

^csimulated used Dow solvent NS-1

Tests also were made of carbon steels formed into small U-bends inside of larger U-bends of 304 stainless steel so that combined galvanic, crevice, and stress corrosion effects could be determined. Couples formed from 1020 steel with 304 stainless and from B-1213 steel with 304 stainless (both at 1:5 area ratios) were exposed to simulated used Dow solvent NS-1 at 250°F for 100 hours and then to deionized, deoxygenated water at 500°F for 200 hours. Corrosion rates were acceptable, crevice attack was minimal, and no stress cracking was apparent, as shown in Table 3.3.3.1.12.

The results of this comprehensive corrosion program show that no severe attack is likely to occur on the carbon steels tested in Dow solvent NS-1 at the times and temperatures projected for the decontamination of Dresden-1. Individual corrosion data sheets are given in Appendix I.

3.3.3.2 Low Alloy Steels (AISI 4142 and 2-1/4 Cr-1 Mo)

General corrosion rates of these low alloy steels in Dow solvent NS-1 usually were higher than those of 1020 carbon steel (Tables 3.3.3.2.1 and 3.3.3.2.2). The corrosion rates of both alloys were reduced, particularly in NS-1 solvent vapor, by a nitrogen atmosphere. Addition of ferric ion increased the corrosion rates of the alloys in liquid NS-1 solvent but appeared to reduce the corrosion rates in NS-1 solvent vapor in the absence of a nitrogen environment (Table 3.3.3.2.3).

Type 4142 low alloy steel with an artificial, TEFLON-produced crevice did not undergo crevice corrosion (Table 3.3.3.2.1). The alloy also did not exhibit crevice attack when galvanically coupled to 304L stainless steel (Table 3.3.3.2.4). Fine pitting, a light form of crevice attack, was observed on the 2-1/4 Cr - Mo alloy galvanically coupled to 304 stainless steel. Crevice corrosion did not appear to be significant.

Both alloys were anodic to 304 stainless steel, and the 2-1/4 Cr - 1 Mo alloy was anodic to 1020 carbon steel. No unusual galvanic effects were noted.

A single U-bend specimen of 4142 alloy exposed to simulated used Dow solvent NS-1 at 250°F for 300 hours showed no indication of stress cracking although etching occurred along the stress bands on the outer surface of the U-bend. Similar results were obtained on single U-bend specimens of both alloys exposed to Dow solvent NS-1 at 275°F for 100 hours. General corrosion rates (Table 3.3.3.2.5) were higher than those of 1020 carbon steel under the same conditions and were not decreased, in one test, by additional inhibitor. Stress etching was observed on the outer surface of the U-bends.

Table 3.3.3.1.12

Galvanic Corrosion Data on Stressed Double U-Bends of Carbon Steels and 304 SS Exposed to Dow Solvent NS-1 with 0.12% Ferric Ion and 0.065% Nickel Ion^a at 250°F for 100 hrs then to Deionized, Deoxygenated Water at 500°F for 200 hrs

<u>Galvanic Double U-Bend^b</u>	<u>Corrosion Rate of Alloy Coupled to 304 SS (mpy)</u>	<u>Stress Results</u>
1020 (1:5 area ratio in favor of 304 SS)	206, 200, 193, 220	No cracking
B-1213 (1:5 area ratio in favor of 304 SS)	553, 467, 481, 393	No cracking

^aSimulated "used" Dow Solvent NS-1.

^bU-bend galvanic couples were made by placing a 3/4" x 3" specimen of carbon steel inside a 2" x 6" 304 SS specimen and bending the specimens simultaneously. The resulting curvature plus the bolting together of the outer 304 SS specimen was sufficient to hold the inner specimen in place.

Table 3.3.3.2.1

General Corrosion Rates of Low Alloy Steels
Exposed to Dow Solvent NS-1 at 250°F for 16 hrs

<u>Alloy</u>	<u>CR (mpy)</u>	<u>Crevice Results</u>
4142	22, 20	
2-1/4 Cr = Mo	155	
4142 with artificial crevice	27	No crevice attack

Table 3.3.3.2.2

General Corrosion Rates of Low Alloy Steel
in Dow Solvent NS-1 Liquid and Vapor at 250°F

<u>Alloy</u>	<u>Phase</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>100-Hour Corrosion Rate (mpy)</u>
4142	Vapor	62	60, 11
4142	Liquid	45	123, 42
4142	Vapor - N ₂ sat'd.	3.9	1.4
4142	Liquid - N ₂ sat'd.	17	157
2-1/4 Cr - 1 Mo	Vapor	43, 66, 100	
2-1/4 Cr - 1 Mo	Liquid	127, 192, 155	
2-1/4 Cr - 1 Mo	Vapor - N ₂ sat'd.	3.5	
2-1/4 Cr - 1 Mo	Liquid - N ₂ sat'd.	86	

Table 3.3.3.2.3

Effect of Ferric Ion on General Corrosion Rates
of Low Alloy Steel in Dow Solvent NS-1 at 250°F

Alloy	Phase	16-Hour Corrosion Rate (mpy)		
		0%	0.1%	0.5%
		Ferric Ion	Ferric Ion	Ferric Ion
2-1/4 Cr - 1 Mo	Vapor - N ₂ sat'd.	70 (avg.)	24	7.0
2-1/4 Cr - 1 Mo	Liquid - N ₂ sat'd.	158 (avg.)	274	363
2-1/4 Cr - 1 Mo	Vapor - N ₂ sat'd.	3.5	15	6.0
2-1/4 Cr - 1 Mo	Liquid - N ₂ sat'd.	86	242	327

Table 3.3.3.2.4

General Corrosion Rates for Galvanic Couples
of Low Alloy Steel with Other Alloys in Dow
Solvent NS-1 at 250°F

<u>Galvanic Couple (1:1 Area Ratio)</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>100-Hour Corrosion Rate (mpy)</u>
4142) 304 SS) Rubber-banded together	191 2.3	
2-1/4 Cr-1 Mo) Rubber-banded 304 SS) together		475 1.6
2-1/4 Cr-1 Mo) Bolted together 304 SS)		440 1.8
2-1/4 Cr-1 Mo) Rubber-banded 304L SS) together	491, 280 2.9, 2.1	
2-1/4 Cr-1 Mo) Rubber-banded 1020) together	124, 92 12, 2.3	

Table 3.3.3.2.5

Single U-bend Corrosion Data on Low Alloy
Steels Exposed to Dow Solvent NS-1 at 275°F

<u>Alloy</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Stress Results</u>
4142	384	No cracking, stress etching
4142	411	No cracking, stress etching
4142 (2x standard inhibitor)	462	No cracking, stress etching
2-1/4 Cr - 1 Mo	451	No cracking, stress etching
2-1/4 Cr - 1 Mo	261	No cracking, stress etching
2-1/4 Cr - 1 Mo	606	No cracking, stress etching
2-1/4 Cr - 1 Mo	581	No cracking, stress etching
2-1/4 Cr - 1 Mo	541	No cracking, stress etching

Decontamination of Dresden-1 with Dow solvent NS-1 at 250°F therefore is not expected to result in substantial corrosion of low alloy steels in the system, particularly in the presence of a N₂ environment. Although low alloy steels appeared to be more susceptible than 1020 steel to light pitting, edge attack, and stress etching, the slight attack is not considered significant because of the short time projected for the cleaning operation. No stress corrosion cracking, crevice corrosion, or significant galvanic corrosion is anticipated. Corrosion test data sheets for the low alloy steels are given in Appendix I.

3.3.3.3 Copper, Nickel and Chromium Based Alloys (CDA 715, Cu:Ni 70:30; CDA 230, Red Brass; CDA 122, Copper; Monel 400; INCONEL 600; INCOLOY 800; INCOLOY 825)

These copper, nickel, and chromium based alloys had excellent corrosion resistance to Dow solvent NS-1 at temperatures and times exceeding those expected in the decontamination of Dresden-1. The alloys had low general corrosion rates, and did not exhibit susceptibility to crevice corrosion, pitting, loss of zinc or nickel, unusual galvanic corrosion effects, or any form of stress corrosion cracking (Table 3.3.3.3.1). No indication of copper redeposition was observed on any specimen.

The corrosion behavior of CDA 715, a 70:30 copper-nickel alloy from which the Dresden-1 system heat exchanger is constructed, was studied in detail. CDA 715 exposed to NS-1 solvent with ferric ion added exhibited an increased corrosion rate but was not subject to crevice attack in the presence of an artificial crevice. (Table 3.3.3.3.2).

Copper alloys and INCONEL 600 exhibited similar corrosion rates when tested in Dow solvent NS-1 as single specimens or when coupled to 300 series stainless steels (Table 3.3.3.3.3). As described previously (Table 3.3.3.1.7) the corrosion rates of 1020 steel were accelerated when the alloy was coupled to INCONEL 600 or INCOLOY 800, but were only slightly accelerated when the alloy was coupled to CDA 715.

Double U-bends of the copper alloys were tested for up to 300 hours at 250°F with no indication of stress cracking (Table 3.3.3.3.4). Figure 3.3.3.3, a metallograph of a section from a single U-bend specimen, shows the absence of both serious attack and stress cracking.

In general, copper alloys and nickel-chromium alloys corroded at average rates of 6-7 mpy and 1 mpy or less, respectively, in NS-1 solvent. Single U-bend specimens of

Table 3.3.3.3.1

General Corrosion Rates of Copper, Nickel, and
Chromium Alloys Exposed to Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>100-Hour Corrosion Rate (mpy)</u>
CDA 715, Cu:Ni 70:30	7.6, 6.0, 4.3	1.9
CDA 230, Red Brass	7.4, 6.5, 8.1	
CDA 122, 99.9 Cu	6.6, 5.2	
MONEL ^a 400	6.4, 6.2	
INCONEL 600	0.57	
INCOLOY 800	0.34, 0.73	
INCOLOY 825	0.72, 1.2	

^aTrademark of Huntington Alloys, Inc.

Table 3.3.3.3.2

General Corrosion Rates of Copper Alloy
CDA 715 in Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>			
	<u>0% Ferric Ion</u>	<u>0.1% Ferric Ion</u>	<u>0.5% Ferric Ion</u>	<u>With Artificial Crevice^a</u>
CDA 715	6.0 (avg)	21	83	6.2

^aNo crevice attack resulted

Table 3.3.3.3.3

General Corrosion Rates for Galvanic Couples
of Copper, Nickel, and Chromium Alloys with
Other Alloys in Dow Solvent NS-1 at 250°F

Galvanic Couple (1:1 Area Ratio)	16-Hour Corrosion Rate (mpy)	100-Hour Corrosion Rate (mpy)
CDA 715) 304L SS) Rubber-banded together	6.4 0.70	
CDA 122) 347 SS) Rubber-banded together		2.6 0.3
CDA 122) 347 SS) Bolted together		3.1 0.4
INCONEL 600) 304 SS) Rubber-banded together		0.4 0.1
INCONEL 600) 304 SS) Bolted together		1.2 0.1

Table 3.3.3.3.4

Stress Corrosion Data for Double U-Bends of
Copper Alloys in Dow Solvent NS-1 at 250°F

Alloy	Test Solution	Exposure (Hours)	Stress Results
CDA 715, Cu:Ni 70:30	Simulated used NS-1	300	No cracking
CDA 230, Red Brass	Dow Solvent NS-1	140	No cracking
CDA 122, 99.9 Cu	Simulated used NS-1	300	No cracking

^aDow solvent NS-1 with 0.12% Fe⁺₃ as ferric ammonium sulfate
and 0.065% Ni⁺₂ as nickel sulfate.

100X

400X

Figure 3.3.3.3

100X and 400X Metallographs of CDA 715 (70:30 Cu:Ni Alloy) exposed to Dow solvent NS-1 for 100 hours at 275°F. Metallograph taken from area of greatest stress on single U-bend specimen. Metal Structure shows no serious attack nor any indication of stress cracking.

copper alloys exposed 100 hours at 275°F gave low corrosion rates and did not exhibit stress cracking, as shown in Table 3.3.3.5. INCONEL corroded at slightly higher rates than the copper alloys did; heat treated INCONEL and INCOLOY corroded at yet higher rates, but these rates are not expected to be unacceptable under actual use conditions. These increased rates were an additional factor in the decision to double the standard inhibitor concentration in NS-1 solvent to provide more protection if the temperature accidentally increases to >250°F during the decontamination.

In summary, no adverse corrosion effects were found on copper, nickel, and chromium based alloys after subjecting them to extensive testing in Dow solvent NS-1 under conditions of time, temperature, and stress exceeding those expected in the actual decontamination of Dresden-1.

3.3.3.4 Specialty Alloys (17-4 P.H., STELLITE #6, HASTELLOY B, and HASTELLOY C)

These alloys exhibited low corrosion rates when exposed to Dow solvent NS-1 for 100 hours at up to 275°F, as shown in Tables 3.3.3.4.1, 3.3.3.4.2, and 3.3.3.4.3. The 17-4 precipitation hardened alloy also had low corrosion rates in tests extended to 360 hours. Other types of corrosion such as crevice attack, excessive galvanic corrosion, or pitting were not observed except for cracking which occurred on the 17-4 P.H. alloy. Some of these data were obtained with single U-bend specimens which also were evaluated for stress corrosion effects.

Galvanic couples of 17-4 P.H. alloy and STELLITE 6 with 304 stainless steel corroded at extremely low rates in NS-1 solvent (Table 3.3.3.4.4). Data for galvanic couples of 1020 carbon steel with 17-4 P.H. alloy and HASTELLOY B were described in Table 3.3.3.1.7. Crevice attack was absent from these specialty alloy specimens formed with crevices in the galvanic couples and underneath the TEFLON washers beneath the bolts holding the ends of U-bends together.

Table 3.3.3.3.5

Single U-Bend Corrosion Data on Copper,
Nickel, and Chromium Based Alloys
Exposed to Dow Solvent NS-1 at 275°F

<u>Alloy and Heat Treatment</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Stress Results</u>
CDA 715, Cu:Ni 70:30 - none	0.62	No cracking
CDA 715, Cu:Ni 70:30 - none	1.8	No cracking
CDA 715, Cu:Ni 70:30 - none	1.1	No cracking
CDA 715, Cu:Ni 70:30 - none	2.0	No cracking
CDA 715, Cu:Ni 70:30 - none	2.1	No cracking
CDA 230, Red Brass - none	0.93	No cracking
CDA 230, Red Brass - none	1.8	No cracking
CDA 230, Red Brass - none	1.3	No cracking
CDA 230, Red Brass - none	2.5	No cracking
CDA 230, Red Brass - none	2.2	No cracking
CDA 122, 99.9 Cu - none	1.5	No cracking
CDA 122, 99.9 Cu - none	1.8	No cracking
CDA 122, 99.9 Cu - none	0.94	No cracking
CDA 122, 99.9 Cu - none	2.4	No cracking
CDA 122, 99.9 Cu - none	2.0	No cracking
INCONEL 600 - none	0.33	No cracking
INCONEL 600 - none	14	No cracking
INCONEL 600 - none	0.29	No cracking
INCONEL 600 - none	12	No cracking
INCONEL 600 - none	8.3	No cracking
INCONEL 600 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	24	No cracking
INCONEL 600 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	31	No cracking
INCONEL 600 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	20	No cracking
INCONEL 600 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	26	No cracking
INCONEL 600 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	25	No cracking

Table 3.3.3.3.5 (Continued)

<u>Alloy and Heat Treatment</u>	<u>100-Hour Corrosion Rate (mpy)</u>	<u>Stress Results</u>
INCOLOY 800 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	65	No cracking
INCOLOY 800 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	69	No cracking
INCOLOY 800 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	62	No cracking
INCOLOY 800 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	59	No cracking
INCOLOY 800 - 2100°F, 1 hr., brine quench, 1200°F, 4 hrs.	49	No cracking

Table 3.3.3.4.1

General Corrosion Rates of Specialty Alloys
Exposed to Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>
HASTELLOY B	2.9
HASTELLOY C	0.64

Table 3.3.3.4.2

Stress and Corrosion Data for Single U-Bends of
Specialty Alloys Exposed to Dow Solvent NS-1 at 275°F

Alloy	100-Hour Corrosion Rate (mpy)	Stress Results
17-4 P.H.	1.9	Cracked
17-4 P.H.	0.55	Cracked
17-4 P.H.	0.56	Cracked
17-4 P.H.	0.79	Cracked
17-4 P.H.	0.49	Cracked
17-4 P.H.)	0.63	Cracked
17-4 P.H.) Double U-Bend	0.71	Cracked
HASTELLOY B	0.82	No cracking
HASTELLOY B	0.62	No cracking
HASTELLOY B	1.0	No cracking
HASTELLOY B	0.89	No cracking
HASTELLOY B	0.98	No cracking
HASTELLOY C	2.5	No cracking
HASTELLOY C	1.7	No cracking
HASTELLOY C	0.36	No cracking
HASTELLOY C	1.1	No cracking
HASTELLOY C	0.41	No cracking

Note: Cracking was typically IGSCC.

Table 3.3.3.4.3

Stress Corrosion Data for Single U-Bends
of Heat-treated 17-4 P.H. Alloy Exposed to
Dow Solvent NS-1 and Deionized Water

Heat Treatment	Test Solution	Test Conditions		Corrosion	Stress Results
		°F	Hrs.	Rate (mpy)	
None	Deionized H ₂ O	275	100 hrs	0.036	No cracking
None	Deionized H ₂ O	275	100	0.12	No cracking
None	Deionized H ₂ O	275	100	0.76	No cracking
None	Deionized H ₂ O, pH 3.6	275	100	0.26	Cracked
None	Deionized H ₂ O, pH 3.6	275	100	0.07	No cracking
900°F - 1 hour	Dow Solvent NS-1	250	100	0.50	Cracked
900°F - 1 hour	Dow Solvent NS-1	250	100	0.92	Cracked
900°F - 1 hour	Dow Solvent NS-1	250	100	1.1	Cracked
900°F - 1 hour	Dow Solvent NS-1	250	100	1.2	Cracked
1150°F-4 hours	Dow Solvent NS-1	250	100	8.8	No cracking
1150°F-4 hours	Dow Solvent NS-1	250	100	4.6	No cracking
1150°F-4 hours	Dow Solvent NS-1	275	100	3.6	No cracking
1150°F-4 hours	Dow Solvent NS-1	275	100	12	No cracking
1150°F-4 hours	Dow Solvent NS-1	250	360	1.3	No cracking
1150°F-4 hours	Dow Solvent NS-1	250	360	2.1	No cracking
1150°F-4 hours	Dow Solvent NS-1	250	360	1.1	No cracking
1150°F-4 hours	Dow Solvent NS-1	250	360	1.5	No cracking

Note: Cracking was typically IGSCC.

Table 3.3.3.4.4

General Corrosion Rates for Galvanic Couples
of Specialty Alloys with 304 SS in Dow Solvent
NS-1 at 250°F

		<u>100-Hour Corrosion Rate (mpy)</u>
17-4 P.H.)	Rubber-banded together	0.2
304 SS)		0.1
17-4 P.H.)	Bolted-together	0.3
304 SS)		0.1
STELLITE 6)	Bolted-together	0.1
304 SS)		0.3

STELLITE 6 was not obtained in a size that would allow it to be tested in a U-bend configuration. HASTELLOY B and C gave no indication of stress cracking at temperatures up to 275°F and for periods up to 300 hours (Tables 3.3.3.4.5 and 3.3.3.4.2).

U-bends fabricated from as-received 17-4 P.H. alloy consistently cracked when tested in NS-1 solvent. Further investigation showed this alloy did not crack in ordinary deionized water but did crack in deionized water when the acidity was decreased to pH 3.6, the pH of Dow solvent NS-1, by adding sulfuric acid. Type 17-4 P.H. alloy, which usually is available in a solution-treated condition, has low ductility, limited formability, and poor resistance to stress cracking unless it is heat treated at 900-1150°F. Therefore, this alloy was heat treated at 900°F for 1 hour or at 1150°F for 4 hours, after which it was descaled in Dow solvent NS-1 and fabricated into U-bends. Specimens subjected to the former heat treatment continued to crack, but specimens heat treated at 1150°F for 4 hours did not crack even after (Table 3.3.3.4.3).

The metallograph in Figure 3.3.3.4.1 shows the crack which developed in the 17-4 P.H. specimen tested in the as-received condition. The metallograph in Figure 3.3.3.4.2 shows the 1150°F heat-treated specimen which did not exhibit cracking. These data point out the importance of properly heat treating the alloy, and suggest that any 17-4 P.H. alloy in Dresden-1 should be inspected after the decontamination and replaced if needed. Single U-bends are more severely stressed than are alloys in actual service; thus the alloy is not expected to undergo unacceptable corrosion in the cleaning operation.

With the exception of 17-4 P.H. alloy in the as-received condition, these speciality alloys did not exhibit significant general weight loss, or galvanic, crevice, or stress corrosion in Dow solvent NS-1 at times and temperatures exceeding those projected for the decontamination. Properly heat treated 17-4 P.H. alloy also did not exhibit adverse corrosion effects. Individual corrosion data sheets are given in Appendix I.

Table 3.3.3.4.5

Stress Corrosion Data for Specialty Alloy
U-Bends Exposed to Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>Specimen Configuration</u>	<u>Test Solution</u>	<u>Exposure (Hrs.)</u>	<u>Stress Results</u>
17-4 P.H.	Single U-Bend	Simulated Used Dow Solvent NS-1 ^a	300	No cracking
HASTELLOY B	Single U-Bend	Simulated Used Dow Solvent NS-1 ^a	300	No cracking
HASTELLOY C	Double U-Bend	Dow Solvent NS-1	140	No cracking

^aDow solvent NS-1 with 0.12% Fe⁺₃ as ferric ammonium sulfate and 0.065% Ni⁺₂ as nickel sulfate.

Figure 3.3.3.4.1

400X metallograph of 17-4 P.H. (as-received) exposed to Dow solvent NS-1 at 275°F for 100 hours. Specimen shows obvious crack which appears to be intergranular.

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Electrolytic
HCl-Methanol Etch

Figure 3.3.3.4.2

400X metallograph of 17-4 P.H. heat treated (1150°F, 4 hours) exposed to Dow solvent NS-1 at 275°F for 100 hours. Metallograph taken from area of greatest stress on single U-bend specimen. Metal structure shows no serious attack and no indication of stress cracking.

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Electrolytic
HCl-Methanol Etch

3.3.3.5 Miscellaneous Alloys (Al 1100, Al 606L, and Titanium)

Dresden-1 is known to contain at least a small amount of aluminum alloy 1100, which is commercially pure aluminum. Its performance was evaluated in Dow solvent NS-1 along with some other aluminum alloys and titanium. General weight loss results are in Table 3.3.3.5.1. Titanium suffered almost no attack, as expected, and the rates of corrosion of aluminum in this weakly acidic solvent were surprisingly low, (approximately 100-200 mpy). Aluminum 1100 exhibited no crevice attack with an artificial crevice present. No unusual galvanic effects occurred when aluminum 1100 was coupled to 304L stainless steel or to 1020 carbon steel (Table 3.3.3.5.2). The corrosion rates of the 1020 steel decreased, but the corrosion rates of aluminum 1100 did not significantly increase.

In summary, a cursory evaluation of these alloys which are present in small amounts in Dresden-1 suggests that they would not suffer serious corrosion when exposed to Dow solvent NS-1.

3.3.3.6 GRAPHITAR 1

The reactor circulation pump, which contains GRAPHITAR 14 bearings, is a key piece of equipment in the decontamination process. Although plans include replacement of the bearings in Dresden-1 after the decontamination is completed, it is important that the bearings remain intact throughout the decontamination. Materials similar to GRAPHITAR, a modified form of graphite, have been known to disintegrate in oxidants such as alkaline permanganate.

Exposure of small pieces of GRAPHITAR 14, alone and coupled with various metals, to Dow solvent NS-1 at 250°F for 100 hours or more produced no deleterious effects. A pump with GRAPHITAR bearings also exhibited no adverse effects in a dynamic loop test of considerably longer duration than that projected for the actual decontamination. This test is described in more detail in Section 3.4.

In summary, unacceptable corrosion effects are not expected to occur on GRAPHITAE bearings exposed to Dow solvent NS-1 during the decontamination of Dresden-1.

Table 3.3.3.5.1

General Corrosion Rates of Miscellaneous Alloys
Exposed to Dow Solvent NS-1 at 250°F

<u>Alloy</u>	<u>16-Hour Corrosion Rate (mpy)</u>	<u>Crevice Results</u>
Al 1100	124, 153	No crevice attack
Al 3003	164, 204	No crevice attack
Al 6061	125, 143	No crevice attack
Ti (.05 Pd)	0.55, 0.64	No crevice attack
Al 1100 with artificial crevice	114	No crevice attack

Table 3.3.3.5.2

General Corrosion Rates Obtained on Galvanic
Couples of Al 1100 With Other Alloys
in Dow Solvent NS-1 at 250°F

<u>Galvanic Couple (1:1 Area Ratio)</u>	<u>16-Hour Corrosion Rate (mpy)</u>
Al 1100) 304 SS) Rubber-banded together	0.49 148
Al 1100) 1020) Rubber-banded together	112 1.8
Al 1100) Bolted together with 1020) TEFLON spacer	153, 163 2.9, 2.7

3.3.4 Effect of Inhibitor Concentration

The inhibitor system used in Dow solvent NS-1 was tested at various concentrations, which in effect measured how changes in the ratio of reactor surface area to solvent volume will affect corrosion rates in Dresden-1. The inhibitor in NS-1 solvent acts via an adsorption mechanism: the more solvent in contact with a given surface area, the more inhibitor available for adsorption onto the surface. When the surface area is large relative to the volume of solvent, more area must be protected but less inhibitor is available at a given concentration.

In a plant such as Dresden-1 the reactor primary system surface to solvent volume ratio varies greatly with the location within the facility and the corrosivity of the solvent should be as insensitive as possible to changes in inhibitor concentration.

The corrosion rates of selected steels and stainless alloys as a function of inhibitor concentration are summarized in Table 3.3.4. The carbon steels were significantly more sensitive to inhibitor concentration than were the stainless alloys. The corrosion rates of 1020 carbon steel averaged 500 mpy in the absence of inhibitor and impurities. With standard inhibitor concentration the corrosion rate decreased to 23-31 mpy while 2X standard inhibitor concentration reduced corrosion rates to 16-27 mpy. The presence of Fe^{+3} and Ni^{+2} in NS-1 solvent also affected the corrosion resistance of carbon steel which exhibited a corrosion rate of 500-600 mpy in the presence of these impurities without inhibitor. The corrosion rates decreased to 250 mpy when the standard concentration of inhibitor was added to NS-1 solvent, and decreased to 50-60 mpy at the 2X standard concentration of inhibitor.

Low alloy steel SA516 corroded at approximately 600 mpy in uninhibited NS-1 solvent, with or without the impurity ions added. The corrosion rate decreased to 390 mpy at the standard inhibitor concentration in the absence of added impurity ions. No reduction in corrosion rate occurred at 2X standard inhibitor concentration in the presence of impurity ions. Apparently this alloy is more sensitive to impurities in the solvent than to inhibitor concentration.

The corrosion performance of stainless alloys was more independent of inhibitor concentration than were the corrosion behaviors of carbon and low alloy steels. The 300 series stainless alloys were passive in both

Table 3.3.4

Effect of Inhibitor in Dow Solvent NS-1 on Corrosion
Rate of Single U-bend Specimens exposed
112 Hours

<u>Alloys</u>	<u>Impurities</u>	<u>Inhibitor Concentration</u>	<u>CR (mpy)</u>	<u>Stress Results</u>
1020	None	0 1/2 C ^c C	510, 497 31, 23 16, 27	No cracking No cracking No cracking
SA516 Gr 70	None	0 1/2 C C	588, 614 423, 410 386, 387	No cracking No cracking No cracking
304 ^a	None	0 1/2 C C	31, 28 2.4, 3.0 2.9, 2.4	No cracking No cracking No cracking
304	None	0 1/2 C C	0.14, 0.07 0.08, 0.09 0.10, 0.07	No cracking No cracking No cracking
304L ^b	None	0 1/2 C C	0.52, 180 2.8, 0.25 0.29, 0.30	No cracking No cracking No cracking
1020	0.12 wt% Fe ⁺³ 0.065 wt% Ni ⁺²	0 1/2 C C	602, 569 244, 288 50, 62	No cracking No cracking No cracking
SA516 Gr 70	0.12 wt% Fe ⁺³ 0.065 wt% Ni ⁺²	0 1/2 C C	609, 623 498, 566 591, 504	No cracking No cracking No cracking
304 ^a	0.12 wt% Fe ⁺³ 0.065 wt% Ni ⁺²	0 1/2 C C	45, 16 5, 4, 23 4.2, 10	No cracking No cracking No cracking
304	0.012 wt% Fe ⁺³ 0.065 wt% Ni ⁺²	0 1/2 C C	2.7, 10 0.64, 0.50 0.44, 0.40	No cracking No cracking No cracking

^aSpecimens were U-bend and then sensitized.

^bExposed at 275°F.

^cC represents recommended - use concentration (i.e. 2Xstandard).

Note: Solvent was saturated with air; temperature was 250°F
unless otherwise specified.

inhibited or uninhibited Dow solvent NS-1. Type 304 stainless steel corroded at <1 mpy with or without inhibitor present and with scale added to the solvent. However, 304 stainless steel specimens, sensitized at 1200°F for 50 hours, exhibited a slightly increased corrosion rate which did not change significantly at varied inhibitor concentrations.

Without inhibitor and before scale was added, sensitized 304 steel corroded at 30 mpy in NS-1 solvent. The corrosion rate decreased to <5 mpy in the presence of both 2X standard and standard concentration of inhibitor. A similar trend was noted in the presence of scale, although the corrosion rates were slightly higher.

Type 304L stainless steel was tested in Dow solvent NS-1 at 275°F and at various inhibitor concentrations. The corrosion behavior of this alloy paralleled that of 304 stainless steel tested at 250°F with one exception: the 304L alloy corroded at 2-10 mpy without inhibitor or scale added, and exhibited decreased corrosion rates of <1 mpy with inhibitor added. When scale was added to uninhibitor solvent at 275°F, one specimen of 304L steel exhibited a much higher corrosion rate than did a duplicate specimen, i.e., 180 mpy vs 0.52 mpy. Previous data (Table 3.3.1.2.3) obtained at two inhibitor concentrations also showed that 304 and 304L stainless steels exhibited sporadic corrosion rates in NS-1 solvent at 275°F, and that the standard inhibitor concentration did not provide adequate protection at this higher temperature. At 275°F the stainless steels can lose passivity and undergo active corrosion unless the inhibitor concentration is maintained at appropriate levels.

All weight loss data reported in Table 3.3.4 were obtained from single U-bend specimens. Examination of the specimens confirmed earlier data which indicated that no stress corrosion is expected to occur on 304 stainless steel that was sensitized after it was fabricated into U-bends.

3.3.5 Effect of Chemical Contaminants

It is a generally accepted concept that a specific chemical or chemical combination will cause stress corrosion cracking in a given alloy under various environmental conditions. This is probably an oversimplification of the problem as the mechanism of stress corrosion cracking does not have an adequate explanation. For example, it is not known why some chemicals will cause stress corrosion cracking while other chemicals, which cause severe surface corrosion, do not.

The necessity of having a specific corrodent in order to produce stress corrosion cracking on a given alloy is a generally accepted but probably an oversimplified concept. A number of different corrodents actually may produce stress attack on an alloy under various environmental conditions. Why certain compounds induce stress cracking whereas other compounds, some of which undergo a vigorous reaction with an alloy, do not induce cracking constitutes a major unanswered question concerning the mechanism of this type of corrosion.

The approach chosen for this study included testing the contaminants which theoretically would be present in Dow NS-1 solvent during the decontamination of Dresden-1 in order to determine the stress cracking effects on these contaminants on 304 stainless steel U-bends and tensile rods.

The impurities of interest were Cl^- , F^- , Pb^{+2} , Fe^{+3} , Cr^{+6} , S^{-2} , Fe_3O_4 , and O_2 . A tensile rod was used to screen potential impurities since a defined amount of cold work and stress could be incorporated into this type of specimen. Specimens were sensitized in two steps by initially heat treating them for six hours at 1250°F and allowing them to cool in the furnace. Subsequently the sensitized specimens were cold worked to a level of 2% or 5% plastic strain and were treated at 1200°F for 50 hours. All heat treatments were carried out in an inert atmosphere. These tensile rod specimens simulated the roll bonded cladding on the pressure vessel of the Dresden-1 reactor in which roll bonding was performed at approximately 1250°F ; stress relief on the finished vessel was provided by a second heat treatment.

Stress tests were carried out under constant strain conditions with a test jig made of 304 stainless steel to avoid galvanic coupling and a Teflon lined spool to prevent interference from other metallic contaminants. Specimens were tested for 100-300 hours at 250°F . Four specimens each were exposed to the impurities as shown in Table 3.3.5.1. A comparison basis was established

Table 3.3.5.1

Stress Tests of 304 SS Tensile Rods Exposed
to Dow Solvent NS-1 with various
Impurity Ions, at 250°F

<u>Preparation of Specimen</u>	<u>Test Conditions</u>	<u>No. Tested</u>	<u>Results</u>
6 hrs. at 1250°F, air cooled, machine pulled to 5% plastic strain, released, stress relieved at 1200°F for 50 hrs. stressed to 2% plastic strain.	300 hrs., air-saturated	16	No failures
"	300 hrs., air-saturated at room temperature, 0.12 wt% Fe ⁺³	4	No failures
"	300 hrs., air-saturated at room temperature, 100 ppm Cl	4	No failures
"	300 hrs., air-saturated at room temperature, 100 ppm Pb ⁺²		
"	300 hrs., air-saturated at room temperature, 0.065 wt% Ni ⁺²	4	No failures
6 hrs. at 1250°F, air cooled, machined, cold worked to 5% plastic strain, released, stress relieved for 30 hrs. at 1200°F in air, stressed to 2% plastic strain	300 hrs., air-saturated at room temperature, heat treatment scale intact from air	4	No failures

Table 3.3.5.1 (continued)

<u>Preparation of Specimen</u>	<u>Test Conditions</u>	<u>No. Tested</u>	<u>Results</u>
6 hrs. at 1250°F, air cooled, machined, cold worked to 5% plastic strain, released, stress relieved at 1200°F for 50 hrs., stressed to 2% plastic strain	200 hrs., air-saturated at room temperature, saturated with Fe_3O_4	4	No failures
Same as above except stressed to 5% plastic strain	"	4	No failures
Same as above except the tensile portion of specimen was surrounded with TEFLON to produce a crevice	"	4	No failures

by preparing 16 tensile rod specimens which were tested using the same conditions but without the impurity in the solvent.

No stress corrosion was found when tensile rod specimens of 304 stainless steel were exposed to Dow solvent NS-1 which contained Fe^{+3} , Cl^- , Pb^{+2} , S^{-2} , Ni^{+2} , or Fe_3O_4 . Specimens with heat treatment scale intact also exhibited no stress corrosion.

Additionally, the corrosive effects of contaminants on double and single two-stage U-bends were investigated as shown in Table 3.3.5.2. This series of tests was conducted on 304 stainless steel which was sensitized before it was fabricated into U-bends. Some specimens were descaled before they were tested.

Table 3.3.5.2

U-Bend Corrosion for Sensitized 304 SS
Exposed at 250°F for 100 Hrs to Dow Solvent NS-1
With Impurity Ions

Additive	Number of Specimens	Results
O ₂ -saturated	2 single 1 double	No cracking No cracking
0.1 wt% H ₂ O ₂	1 single 1 double	No cracking No cracking
Air-saturated Fe ⁺³ as Fe(NH ₄)(SO ₄) ₂	2 singles (descaled) 3 doubles (descaled)	No cracking No cracking
Air-saturated 0.05 Fe ⁺³ as Fe(NH ₄)(SO ₄) ₂	2 singles (descaled) 2 doubles (descaled)	No cracking No cracking
Air-saturated 0.12 Fe ⁺³ as Fe(NH ₄)(SO ₄) ₂	3 singles (descaled) 2 doubles (descaled)	No cracking One cracked one did not
Air-saturated 100 ppm F ⁻	3 single (descaled) 3 doubles (descaled)	No cracking No cracking
Deaerated 100 ppm F ⁻	1 single (descaled) 2 doubles (descaled)	No cracking No cracked
Air-saturated 10 ppm F ⁻	1 single (descaled) 2 doubles (descaled)	No cracking No cracking
Deaerated 10 ppm F ⁻	1 single 1 double	No cracking No cracking
Air-saturated 100 ppm Cl ⁻	2 singles (descaled)	No cracking

Specimens were sensitized at 1200°F for 50 hours, and were not descaled unless otherwise indicated.

Table 3.3.5.2 (Continued)

Additive	Number of Specimens	Remarks
Deaerated 10 ppm F^-	1 single 1 double	No cracking No cracking
Air-saturated 100 ppm Cl^-	2 singles (descaled) 2 doubles	No cracking No cracking
Deaerated 100 ppm Cl^-	2 singles 1 double	No cracking No cracking
Air-saturated 10 ppm Cl^-	2 singles 1 double	No cracking No cracking
Air-saturated 100 ppm Pb^{+2} as lead acetate	5 doubles (descaled)	No cracking
Air-saturated 100 ppm S^{+2} as $Na_2S \cdot 9H_2O$	5 doubles (descaled)	No cracking
0.1 wt% Cr^{+6} as K_2CrO_4 air saturated	2 singles (descaled) 2 doubles (descaled)	No cracking One cracked one did not
"	2 singles (descaled) 2 doubles (U-bent then sensitized)	No cracking
0.1 wt% Cr^{+6} as K_2CrO_4 Air-saturated	2 singles 1 double	No cracking Cracked
10 ppm Cr^{+6} as K_2CrO_4 Air-saturated	2 singles (descaled) 2 doubles	No cracking No cracking

Specimens were sensitized at 1200°F for 50 hours, and were not descaled unless otherwise indicated

Note: When cracking occurred it was IGSCC.

Increasing the oxidizing power of Dow solvent NS-1 by saturating it with air or by adding H_2O_2 did not appear to increase the tendency of the solvent to cause stress corrosion of the specimens. Ferric ion caused stress attack at concentrations of 0.12 wt% but not at lower concentrations. These data are consistent with those discussed in Section 3.3.1.5 where Fe^{+3} at a concentration of 0.12 wt% caused stress cracking on highly sensitized 304 stainless steel. However, Fe^{+3} at this concentration also produces stress attack on sensitized 304 stainless steel exposed to deionized water under similar conditions. Slow strain rate testing at iron levels of 1200 ppm did not show evidence of accelerated failure.

Single and double U-bend specimens exposed to deaerated and air-saturated NS-1 solvent which contained 100 ppm F^- or Cl^- did not exhibit cracking. Pb^{+2} and S^- at 100 ppm also did not induce adverse corrosion effects.

When tested as a possible inhibitor of intergranular stress corrosion, hexavalent chromium at a concentration of 1000 ppm did not act as an inhibitor for corrosion of sensitized 304 stainless steel which contained heat scale, and induced IGSCC on descaled coupons. No attack was evident in the presence of 10 ppm Cr^{+6} . This corrosion effect is not expected to cause adverse conditions during the decontamination of Dresden-1 since NS-1 solvent does not have the ability to oxidize chromium to a valence of +6.

Specimens of 304 stainless steel were sensitized for 50 hours at 1200°F and were fabricated into extra-wide double U-bends. These specimens exhibited corrosion rates of 1-4 mpy and showed no stress attack when exposed for 112 hours at 250°F to Dow solvent NS-1 saturated with magnetite (5g Fe_3O_4 /100 ml solvent) as shown in Table 3.3.5.3.

In summary, screening of impurities in Dow solvent NS-1 for their adverse effects on the corrosion resistance of 304 stainless steel showed that only Fe^{+3} at concentrations of >1200 ppm appeared to promote intergranular stress corrosion. Because much of the radioactive scale in Dresden-1 consists of iron, the concentration of iron in NS-1 solvent can be expected to approach 1200 ppm when the primary reactor is decontaminated. However, results from tensile rod and wide U-bend tests, and WOL fracture tests, lead to the following conclusions:

1. Fe^{+3} at concentrations (1200 ppm) which cause intergranular stress attack on sensitized 304 stainless steel will produce this attack in deionized water as well as in Dow solvent NS-1.

Table 3.3.5.3

Corrosion Results for Wide Double U-Bends
Exposed to Dow Solvent NS-1
Saturated with Fe₃O₄.

<u>Corrosion Rate (mpy)</u>	<u>Stress Results</u>
2.5	No stress cracking
2.2	No stress cracking
1.7	No stress cracking
2.0	No stress cracking
3.5	No stress cracking
3.8	No stress cracking
1.6	No stress cracking
1.6	No stress cracking
2.4	No stress cracking
2.6	No stress cracking
1.7	No stress cracking
2.1	No stress cracking
2.5	No stress cracking
2.3	No stress cracking
0.72	No stress cracking
0.80	No stress cracking

Conditions

Temperature: 250°F

Time: 112 hours

Specimens: 6"X2"C0.063". U-bend, then sensitized
at 1200°F for 50 hours

Solvent: 5 g Fe₃O₄/100 ml NS-1

2. Only the most severely stressed type of 304 stainless steel will undergo attack at 1200 ppm Fe^{+3} in NS-1 solvent.
3. Only U-bends which are fabricated from 304 stainless steel sensitized for >30 hours will exhibit attack at high concentrations of iron in NS-1 solvent.

3.3.6 Effect of Exposure Temperature

Dresden-1 will be chemically cleaned with Dow solvent NS-1 at 250°F, a temperature high enough to allow rapid dissolution of the mixed oxide scale (nickel ferrite) in the primary system but not so high as to thermally degrade the solvent. Appreciable thermal decomposition of the solvent does not occur until the temperature is greater than 300°F. Therefore, the major concern regarding small temperature transients is the increase in corrosion rate that occurs at higher temperatures. The engineering aspects of the cleaning will allow thermal transients to be limited to $\pm 25^\circ\text{F}$. Thus, in corrosion tests the highest temperature chosen for study was 275°F.

General corrosion tests at 275°F on various heats of type 304 stainless steels showed that to be effective the inhibitor system must be maintained at a specified concentration (Section 3.3.1.2 and Table 3.3.1.2.3). If the inhibitor is maintained at the recommend concentration, protection from general corrosion is complete up to 275°F. Additional work was done to assess the stress corrosion characteristics of a variety as two-stage single U-bends, at 275°F (Table 3.3.6).

No stress corrosion was observed on single U-bend specimens exposed to Dow solvent NS-1 for 112 hours at 275°F instead of at the usual 250°F. The inhibitor was effective, and the solvent was thermally stable at 275°F.

Adverse corrosion effects therefore are not expected when Dresden-1 is decontaminated with Dow solvent NS-1 under the planned cleaning conditions if brief temperature excursions to 275°F occur.

Another aspect of the temperature performance of NS-1 solvent concerns the fate of solvent sequestered in crevices of 304 stainless steel in the primary reactor after it is decontaminated. Trapped solvent which is not removed by subsequent flushing operations will thermally decompose when the plant is returned to operating temperatures. Therefore the effects of solvent degradation products on the corrosion of 304 stainless steel crevices were determined.

As described in section 3.3.1.6 under WOL fracture mechanics tests, highly stressed fatigue cracks were induced in sensitized 304 stainless steel specimens

Table 3.3.6

Single U-Bend Stress Tests of Alloys Exposed
to Dow Solvent NS-1 at 275°F for 112 Hours

<u>2-Stage Single U-Bend Specimens</u>	<u>Number Tested</u>	<u>Results</u>
304	14	No cracking
304 sensitized (1200°F, 50 hrs), descalded, U-bent	22	No cracking
304L	17	No cracking
304L sensitized (1200°F, 50 hrs), descalded, U-bent	15	No cracking
316	5	No cracking
17-4 P.H. heat treated (1150°, 4 hrs)	5	No cracking
HASTELLOY B	5	No cracking
INCONEL 600	5	No cracking
INCONEL 600 sensitized (1200°F, 50 hrs), U-bent	5	No cracking
INCOLOY 800 sensitized (1200°F, 50 hrs), U-bent	5	No cracking
405	5	No cracking
405 temper-embrittled	5	No cracking
410	5	No cracking
416	5	No cracking
446	5	No cracking
446 temper-embrittled	5	No cracking
442 temper-embrittled	5	No cracking
2-1/4 Cr - 1 Mo	5	No cracking
4142	5	No cracking
1020	5	No cracking
Red Brass, CDA 230	5	No cracking
70/30 Cu/Ni, CDA 715	5	No cracking
MONEL	5	No cracking

and solvent was allowed to decompose in the crevices which were under tensile loading. No adverse corrosion effects were indicated by this testing.

In the second approach, solvent was trapped in a dead-end area of sensitized 304 stainless steel which was under high residual stress. A specimen which could be examined optically and metallographically after it was exposed to NS-1 solvent was designed so that the favorable results obtained in earlier fracture mechanics tests could be evaluated further.

A 304 stainless steel specimen with two types of crevices was fabricated from standard tubing equipped with a SAGELOK fitting and was sensitized. The geometry of this couple was such that the extensive machining required for its fabrication caused high residual stresses to be present in the assembled specimen (Figure 3.2.3.3.1). It was assembled while it was submerged in the test solution, and was mechanically tightened 1-1/4 turns past hand-tightness. Each specimen was exposed to Dow solvent NS-1 for 100 hours at 250°F, and was transferred unrinsed to an autoclave that contained deoxygenated, deionized water (O_2 <0.3 ppm) for 100 hours at 500°F, a temperature above which the solvent decomposes. After the test each specimen was disassembled and examined; Figure 3.3.1.4.9 shows a typical fitting. No evidence of adverse corrosion was found on any of four specimens tested in this manner.

Samples of Dow solvent NS-1 also were thermally degraded at 500°F with metal coupons present in order to give breakdown products for analysis. Infrared spectral analysis showed carbon dioxide as the principal breakdown product along with methane, ammonia, ethylene and propylene produced in decreasing quantities in the order listed. Carboxylic acid salts and inorganic sulfates were the main components remaining in the water solution. The infrared spectrum of a small acetone and water-soluble residue indicated the presence of methylene groups, carboxylic acid salts, and a small amount of inorganic salts. Prolonged heating above the decomposition temperature (300-350°F) of NS-1 solvent probably would produce diethylamine, triethylamine, and diamines.

In summary, single U-bend specimens of various alloys did not exhibit stress corrosion when exposed to Dow solvent NS-1 at 275°F for 112 hours. Corrosion also did not occur on sensitized 304 stainless steel specimens which contained a complex crevice configuration when the specimens contained trapped NS-1 solvent and were

further exposed to NS-1 solvent in an autoclave. Finally, the products from the thermal decomposition of NS-1 solvent were mainly simple organic molecules and small amounts of inorganic salts. These breakdown products are volatile enough to be carried over with the steam in Dresden-1 or can be removed by a polishing treatment.

3.4 DYNAMIC TEST LOOP RESULTS

A dynamic test loop was designed and constructed to evaluate the behavior of materials in Dow solvent NS-1 as a function of solvent velocity. The primary objective was to test GRAPHITAR 14, the material from which the reactor circulation pump bearings in Dresden-1 are constructed. The secondary objective was to test bimetallic welds and general corrosion specimens. Monitoring Equipment, based on a linear polarization technique, was used to obtain instantaneous corrosion rates. Initially GRAPHITAR 14 bearings in the stainless steel immersion pump of the loop were evaluated in a continuous 300 hour test with NS-1 solvent. The pump design was similar to that of the main coolant pumps at Dresden-1. All internal surfaces of the pump were inspected visually before this test and were photographed; the thicknesses of the materials to be wetted by the solvent were measured. The conditions of the test were chosen so as to closely simulate the proposed cleaning conditions. The temperature was controlled at 250°F $\pm$ 10°F; flow rates were 7 gal/min through the 2" diameter piping of the test loop. The solvent initially was saturated with air; depleted air was not replenished during the test.

The pump was removed from the loop after 300 hours, and was disassembled and re-examined. All surfaces were shiny, and did not exhibit wear as determined by measurements of bed surfaces. These findings were documented with photographs, some of which are found at the end of this section. Photographs taken at the conclusion of the 300 hour test are not shown because of the absence of changes in the pump surfaces. However photographs taken after the pump was operated for 1000 hours are given later in this section, and can be compared with the initial photographs.

The pump was reinstalled in the loop, and 300 hour weight loss tests were carried out on welded couples, stressed U-bends and tensile rods, bimetallic crevice

specimens, and general weight loss specimens. In addition, two TEFLON holders as shown in Figure 3.2.5.5 were fabricated and inserted into the dynamic test loop piping. Each hollow holder fit snugly into the piping so that all solvent passed through the center of the holder rather than around the exterior of the holder. Stainless steel specimens, placed parallel to the flow path of the solvent, were contained in the TEFLON holders in order to give some measure of the effects of solution velocity on the corrosion rates of the alloys. All specimens were exposed for 300 hours at 250°C to Dow NS-1 solvent which contained $>0.12 \text{ wt\% Fe}^{+3}$. The solvent was saturated with air at the beginning of the test and again after the test had proceeded for 150 hours.

The results of the weight loss tests are given in Table 3.4.1. Welded galvanic couples of the various stainless steel alloys exhibited low corrosion rates in Dow solvent NS-1. Specimens were coupled at area ratios of 1:1 except for specimens in which 304 stainless steel was welded to a carbon steel or was coupled with GRAPHITAR 14. Stainless alloys coupled to sensitized 304 stainless steel all exhibited similar corrosion rates of 23-31 mpy, as did presensitized 304 stainless steel welded to itself.

No adverse weld attack occurred on any of the welded stainless couples although HASTELLOY B coupled to 304 stainless steel produced slightly more weight loss on the 304 member of the couple (41 mpy) than did the other stainless alloys. This result was not unexpected since HASTELLOY B is a more noble alloy in the solvent than are the 300 series stainless alloys (see Section 3.3.3.1.7 for general corrosion rates of HASTELLOY B).

Although GRAPHITAR 14 might be expected to form a galvanic couple with 304 stainless steel, no such coupling effects were found when the 304 alloy was coupled to GRAPHITAR 14 at a 2:1 area ratio and exposed to Dow solvent NS-1. Galvanic rates remained low on both the stainless steel (1-3 mpy) and GRAPHITAR 14 (+0 mpy). GRAPHITAR 14 gained a slight amount of weight due to absorption of water, a normal behavior which caused no physical property changes.

A galvanic couple of SA 516 Gr 70 alloy and 304 stainless steel, at a 20:1 area ratio, was fabricated to simulate a cladding crack in the reaction pressure vessel for

Table 3.4.1

Corrosion Data from Dynamic Test Loop

<u>Specimen</u>	<u>Corrosion Rate (mpy)</u>	<u>Stress Result</u>
<u>Galvanic Couples (Welded 1:1 area ratio unless otherwise noted)</u>		
410 ^a - 304 Sensitized (1200°F, 50 hrs)	27 (avg. of 6)	No cracking
316 - 304 ^a Sensitized (1200°F, 50 hrs)	23 (avg. of 5)	No cracking
304 - 304 Sensitized (1200°F, 50 hrs)	31 (avg. of 5)	No cracking
316 - 316	3.7, 4.4	No cracking
304 Sensitized - 304 Sensitized (1200°F, 50 hrs)	28.5, 28.6	No cracking
304 ^a - HASTELLOY B	41	No cracking
304 - SA 516 Gr 70 (20:1 area ratio; welded with crevice to simulate cladding crack)	1756	
304 ^a - GRAPHITAR 14 (2:1 area ratio, bolted together)	3, <1	
<u>General Weight Loss</u>		
304 (U-Bends)	12	No cracking
STELLITE 6	2	
446	0.02 (avg. of 3)	
446 (stagnant Conditions)	1-3	
410	1-6 (avg. of 3)	
410 (stagnant conditions)	3-5	

^aAnodic member

Table 3.4.1 (continued)

<u>Specimen</u>	<u>Corrosion Rate (mpy)</u>	<u>Stress Results</u>
<u>Crevice Specimens (4" X 4") plate with TEFLON crevice)</u>		
304	1	(no crevice attack)
MONEL 400	3-6	(no crevice attack)
INCOLOY 800	7	(no crevice attack)

Stress Specimens

Sensitized, cold worked tensile rods, 1250°F for 6 hours, strained 5%, 1200°F for 50 hours, stressed to 2% strain

No cracking

Conditions

Time: 300 hours

Temperature: 250°F

Flow rate: 7.5 ft/sec. unless specified otherwise

Solution: simulated used Dow solvent NS-1 with 0.12-0.20 wt% Fe; initially air-saturated, and then saturated with oxygen after 150 hours

Dresden-1. A 3" X 3/4" piece of SA 516 Gr 70 was welded to a 12" X 6" piece of 304 steel. The weld was made on three sides, leaving one long seam exposed to create an open crevice between the metals. Corrosion rates of the carbon steel were high (1756 mpy) when the couple was exposed to Dow solvent NS-1 in the dynamic test loop, but the weight loss was uniform. No evidence of crevice corrosion was found, with attack resulting solely from the galvanic coupling between the two alloys.

General weight loss specimens of STELLITE 6 and CN-7M cast stainless alloy (Alloy 20) corroded at low rates of 1-2 mpy. Specimens of 410 and 446 stainless alloys were tested as weight loss coupons on the stainless steel specimen holder and in the TEFLON high velocity holder. These alloys appeared to corrode at lower rates under conditions of higher solution velocity: i.e., under stagnant conditions 410 and 466 alloys corroded at rates of 3-5 mpy and 1-3 mpy respectively; at 7.5 ft/sec. solution velocity the alloys corroded at rates of 1.6 mpy and 0.02 respectively.

Single, unsensitized U-bends of 304 stainless steel did not exhibit crevice corrosion when exposed to NS-1 solvent. Specimens consisting of 4" X 4" flat plates of 304 stainless steel, MONEL 400, or INCOLOY 800 separated by a multi-creviced TEFLON spacer also did not undergo crevice corrosion in NS-1 solvent, and exhibited low general corrosion rates.

Sensitized and cold worked tensile rod specimens of 304 stainless steel showed no evidence of stress corrosion in the dynamic loop tests.

In summary, corrosion tests of various alloys and specimen configurations showed that exposure of the specimens to Dow solvent NS-1 in a dynamic test loop had no apparent adverse effects compared to tests performed under static conditions.

In additional tests, probes of AISI type 1010 carbon steel, CDA 122 copper, aluminum alloy 1100, and 304 stainless steel were inserted into the dynamic test loop in order to measure corrosion rates by linear polarization techniques. These probes represented the classes of alloys present in Dresden-1.

The purpose of this test was to obtain two types of information:

1. Comparison of electrochemical corrosion rates with weight loss corrosion rates of the alloys, and
2. measurement of the reactions of the alloys to changes in their chemical environment.

The difference between the way in which weight loss corrosion rates and electrochemical corrosion rates are determined should be considered when comparing these types of rates. Weight loss corrosion rates are determined by measuring a weight decrease over a time interval:

$$CR = K \frac{\Delta w}{\Delta t}$$

where CR is the corrosion rate in mpy,
 Δw is the weight loss
 Δt is the exposure time, and
 $K = 534/\rho a$

(where ρ is the density and
 "a" is the area of the specimen).

In contrast, an electrochemical corrosion rate is measured instantaneously (CR inst.), and is not an average value. Therefore,

$$CR = K \frac{\Delta w}{\Delta t} = \frac{1}{t} \int_0^t CR \text{ inst. } X dt$$

Since electrochemical monitoring instantaneously senses corrosion rates, these rates are likely to reflect changes in the chemical environment of an alloy. The effects of changes in solvent temperature, flow rate, and oxygen content on the instantaneous corrosion rates of the alloys were determined with the dynamic loop as shown in Figure 3.4.1. During the part of the tests period from 70-200 hours the loop was operated so as to simulate the conditions planned for the decontamination of Dresden-1. The temperature and flow rate were maintained at 250°F and 6 gal/hour, respectively while the solution was blanketed with nitrogen. The electrochemical corrosion rates of the alloys were in agreement with those determined by weight loss techniques (Sections 3.3.1 and 3.3.3), and averaged, 2.5 mpy for 304 stainless steel, 80 mpy for aluminum alloy 1100, 50 mpy for AISI type 1010 carbon steel, and 20 mpy for copper.

Although corrosion rates of the alloys were low at the beginning of the test, the rates increased as the temperature was increased to 160°F, with carbon steel showing the largest increase in corrosion rate. With this temperature maintained, the corrosion rates decreased to a lower equilibrium value when the temperature was subsequently increased to 230°F, the corrosion rates for all the alloys again increased with the aluminum alloy exhibiting the largest increase.

After approximately 250 hours of elapsed time the flow rate of NS-1 solvent was increased from 6 gal/min. to 22.5 gal/min., with all alloys but copper undergoing increased corrosion rates. As the flow rate was decreased from 22.5 gal/min. to 15 gal/min., the rate of decrease in corrosion corresponded to the change in velocity.

Addition of oxygen to the solvent 350 hours into the test significantly increased the corrosion rate of copper and 1010 steel, but did not adversely affect the performance of the aluminum or stainless alloys.

The results from this single test indicated that the alloys studied did appear to behave electrochemically in a manner that was predictable from weight loss tests. Properly selected alloy probes might be used to monitor transient conditions during the decontamination of Dresden-1. Understanding the way in which different alloys react to the transient conditions would allow the effects of the conditions to be predicted. Much additional research would be needed, but the ground-work has been laid for future work in this area.

Following the electrochemical corrosion testing, the immersion pump was removed from the loop and disassembled. Comparison of photographs (Figures 3.4.2 through 3.4.7) taken before the test and after approximately 1000 hours showed that Dow solvent NS-1 was compatible with the 316 stainless steel and GRAPHITAR 14 parts in the pump. Remeasurement of the dimensions of the pump and the GRAPHITAR 14 bearings also showed that no significant change took place in the internal parts of the pump (Table 3.4.2).

Figure 3.4.3

Immersion pump rotor exposed to Dow solvent NS-1 for approximately 1000 hours under various conditions.

Figure 3.4.4

Rear bearing of immersion pump before it was exposed to Dow solvent NS-1.

Figure 3.4.5

Rear bearing of immersion pump exposed 1000 hours to Dow solvent NS-1 in dynamic test under various conditions.

Figure 3.4.6

Impeller of immersion pump before it was exposed to Dow solvent NS-1.

Figure 3.4.7

Impeller of immersion pump exposed approximately 1000 hours to Dow solvent NS-1 under various conditions.

Table 3.4.2

Dynamic Loop Test of Pump Bearing

	<u>Diameter (inches)</u>
<u>Initial Pump Dimensions:</u>	
Impeller	5.002
Front Bearing	0.9188 (inside)
Back Bearing	0.9188 (inside)
Rotor	
Back Shaft	0.9145 (outside)
Front Shaft	0.9147 (outside)
<u>Final Pump Dimensions:</u> (After 1,000 Hours)	
Impeller	5.002
Back Bearing	0.9200
Front Bearing	0.9190
Rotor	
Back Shaft	0.9145 (outside)
Front Shaft	0.9145 (outside)

Note: GRAPHITAR 14 gained weight because it absorbed water when exposed to NS-1 solvent, a characteristic which does not affect its performance as a bearing material.

3.5 Electrochemical Studies - Ohio State Report

Commonwealth Edison requested that Prof. Roger Staehle of the Ohio State University provide supporting experimental studies and consultation in connection with the materials evaluation program for decontaminating Dresden-1. The studies by Prof. Staehle were intended to be basically electrochemical and to complement the corrosion study carried out at Dow. Instead of covering a wide range of alloys, the electrochemical study would concentrate on type 304 stainless steel, the most abundant alloy at Dresden-1.

Type 304 stainless steel was evaluated in Dow solvent NS-1 via potential vs. time plots, anodic and cathodic polarization studies, and slow strain rate measurements with and without potential control. These studies were carried out over a wide range of environmental conditions.

Conclusions from the Ohio State University electrochemical studies on Dow solvent NS-1 were:

1. Evaluation of the polarization behavior of 304 stainless steel in the solvent is complicated because the solvent undergoes oxidation which partly obscures that being measured for the metal. Therefore, the metal cannot be evaluated over the full range of anodic potentials. Despite this difficulty, certain important conclusions from the results of polarization study were obtained:
 - a. Dow solvent NS-1 did not break down the protective film on 304 stainless steel.
 - b. The oxidation rate of the metal observed in the electrochemical tests corresponded well with the weight loss behavior observed in corrosion tests at Dow.
 - c. No significant difference was found between the performance of sensitized and nonsensitized 304 stainless steel in NS-1 solvent.
 - d. The absence of the inhibitor from Dow solvent NS-1 did not seem to significantly change the reactivity of the stainless steel.
 - e. Stress corrosion cracks are not expected to occur to any significant extent.
2. The slow strain rate stress corrosion cracking studies were conducted over a range of temperatures, electrochemical conditions, and metallurgical conditions. No evidence of susceptibility to stress

corrosion cracking was found under conditions expected during the decontamination process, especially as shown by fractographic examinations.

3.6 PLATING OF COPPER FROM DOW SOLVENT NS-1 EXAMINATION OF DRESDEN-1 SCALE AND SLUDGE SAMPLES SHOWED THAT AN ESTIMATED 30-50 LBS OF COPPER IS PRESENT IN THE SYSTEM.

This estimate is rather indefinite because the test deposits varied widely in copper content.

The copper present in the scale and sludge presents a potential plating problem. Laboratory tests indicated that copper will plate from Dow NS-1 onto 304 stainless steel coupled to 410 or 405 stainless steel, and onto uncoupled 304 stainless steel if it is exposed for extended periods of time.

The configuration of the specimen used for laboratory testing is shown in Figure 3.6.1. Copper oxide was placed in the crevice region of the galvanic couple, the area most likely for plating to occur. Couples of 405 stainless steel with 304 stainless steel were used for all tests since this type of bimetallic junction is the one most likely to be exposed to cleaning solvent at Dresden-1.

Initial tests with copper at concentrations of 500 and 1000 ppm Cu^{+2} , as CuO , both with and without ferric ion present, showed that plating from NS-1 solvent occurred on the couples exposed 100 hours at 250°F. Additional tests were performed to determine the amount of time which would elapse before plating would occur.

Since copper sulfate dissolved in the test solution faster than CuO , copper sulfate was used in the remaining tests. No plating occurred in four or 16 hour tests in which specimens were exposed at 250°F to NS-1 solvent which contained 1000 ppm Cu^{+2} as CuSO_4 and 1200 ppm Fe^{+3} as $\text{Fe}(\text{NH}_4)(\text{SO}_4)_2$. Specimens exposed 24 hours exhibited a noncollectable, light deposit or "flash" of copper.

An attempt was made to establish a threshold concentration for plating of copper. When copper was added at concentrations of 50, 100, and 500 ppm, as CuSO_4 to Dow solvent NS-1 which also contained 1200 ppm Fe^{+3} as $\text{Fe}(\text{NH}_4)_2$, plating occurred after 100 hours on all specimens exposed.

Figure 3.6.1

Test Configuration for Plating of Copper
From Dow Solvent NS-1 in Laboratory

The only encouraging result from the laboratory data was that the time necessary for initiation of copper plating appeared to be in excess of 16 hours. Apparently the solvent complexed the copper and held it in solution, and some time was required before the complex disassociated and the copper plated on the test alloys. The stainless alloys were highly corrosion resistant in Dow solvent NS-1, and apparently required extended exposure times before enough copper displaced iron to produce a flash deposit. Under these conditions, electrolytic filtration probably could be used to remove the copper. A large-scale experiment had to be performed before this could be attempted in order to verify that plating would not occur before some period such as 16 hours elapsed.

A dynamic test was designed to utilize larger specimens of 304 and 405 stainless steels coupled in a crevice configuration since laboratory tests established that the most heavily plated regions occurred in crevice areas. The specimens were placed in cannisters which were part of the dynamic test loop (Figure 3.6.2).

The specimens were isolated from the test loop as shown in Figure 3.6.3. The specimen holders were made so that solution flowed past the specimens without turbulence, but with a velocity sufficient to ensure a constant concentration of copper in solution. A 100 ppm copper solution was used for this test since the analysis of scales from Dresden-1 indicated this amount of copper was present in the Dresden-1 system.

Three specimen holders were placed in parallel so that two specimens could remain on test while one was removed for examination. As shown in Figure 3.6.4, specimens exposed in the test loop for 5.5 hours exhibited a small flash of copper on the crevice side but not on the outer surfaces. A specimen examined after 16 hours exhibited a more uniform flash of copper in the crevice region as well as the beginning of a flash outside the crevice (Figure 3.6.5). A duplicate specimen removed at 16.5 hours confirmed these results (Figure 3.6.6).

A specimen removed at 21 hours exhibited a pronounced flash of copper inside the crevice, and the beginning of copper plating on the outer surfaces (Figure 3.6.7); the specimens removed at 22.5 hours were similar in appearance, indicating the results are predictable (Figure 3.6.8). The specimen tested for 24 hours exhibited heavy plating inside the crevice region and the beginning of plating on the outside surfaces (Figure 3.6.9).

Figures 3.6.10 through 3.6.15 exhibit the results of the copper plating tests for other similar test conditions.

Figure 3.6.2

Holder for specimens used to evaluate
plating of copper from Dow solvent NS-1
in dynamic loop tests.

Figure 3.6.3

High Pressure Circulation Loop

Figure 3.6.4

Copper plating specimens after 5.5 hours in Dow solvent NS-1 (250°F) with 100 ppm cupric ion.

Note: A limited number of copies of this report were made with color reproductions of the original photograph. The remaining copies show black and white reproductions.

Figure 3.6.5

Copper plating specimens after 16 hours in Dow solvent NS-1 (250°F) with 100 ppm cupric ion.

Note: A limited number of copies of this report were made with color reproductions of the original photograph. The remaining copies show black and white reproductions.

Figure 3.6.6

Copper plating specimens after 16.5 hours in Dow solvent NS-1 (at 250°F) with 100 ppm cupric ion.

Note: A limited number of copies of this report were made with color reproductions of the original photograph. The remaining copies show black and white reproductions.

Figure 3.6.7

Copper plating specimens after 21 hours in Dow solvent NS-1 (250°F) with 100 ppm cupric ion.

Note: A limited number of copies of this report were made with color reproductions of the original photograph. The remaining copies show black and white reproductions.

Figure 3.6.8

Copper plating specimens after 22.5 hours in Dow solvent NS-1 (250°F) with 100 ppm cupric ion.

Note: A limited number of copies of this report were made with color reproductions of the original photograph. The remaining copies show black and white reproductions.

Figure 3.6.9

Copper plating specimens after 24 hours in Dow solvent NS-1 (250°F) with 100 ppm cupric ion.

Note: A limited number of copies of this report were made with color reproductions of the original photograph. The remaining copies show black and white reproductions.

Figure 3.6.10

Copper plating specimens after 5.5 hours in Dow Solvent NS-1 (250°F) with 100 ppm cupric ion. (Outside Crevice)

Figure 3.6.11

Copper plating specimens after 16 hours in Dow Solvent NS-1 (250°F) with 100 ppm cupric ion. (Outside Crevice)

Figure 3.6.12

Copper plating specimens after 16.5 hours in Dow Solvent NS-1 (250°F) with 100 ppm cupric ion. (Outside Crevice)

Figure 3.6.13

Copper plating specimens after 21 hours in Dow Solvent NS-1 (250°F) with 100 ppm cupric ion. (Outside Crevice)

Figure 3.6.14

Copper plating specimens after 22.5 hours in Dow Solvent NS-1 (250°F) with 100 ppm cupric ion. (Outside Crevice)

Figure 3.6.15

Copper plating specimens after 24 hours in Dow Solvent NS-1 (250°F) with 100 ppm cupric ion. (Outside Crevice)

The velocity of the solution was maintained at approximately 1-2 ft/sec. to prevent turbulence from cleaning the outside surfaces of the specimens.

This experiment indicated that copper does not plate from Dow solvent NS-1 onto the stainless alloys tested until 16-21 hours have elapsed, but that plating will occur in crevice regions in the relatively short time of five hours. These dynamic test results which verified the earlier laboratory results indicated that it should be possible to remove the copper from solution before plating can occur over general surface areas.

A small electrolytic cell was built to measure the rate at which copper plates from Dow solvent NS-1. A cell with a packed GRAPHITE bed was chosen since it presents a high cathode surface area to the solution, enabling low concentrations of copper to be plated at a reasonable rate. In this cell, copper plated from NS-1 solvent at high efficiency, with minimum liberation of hydrogen at the cathode, but the anodic reaction liberated oxygen at unacceptable levels.

This problem was overcome by using a unique proprietary anode which allowed removal of oxygen while the cell was operated at 250°F. A laboratory model of this cell operated satisfactorily. A large-scale cell was constructed and sent to Dresden-1 for use during cleaning of the General Electric corrosion test loop.

The copper concentration was 35 ppm before the cell was turned on during the corrosion test loop cleaning, but decreased to <1 ppm within two hours after the cell was activated. The copper concentration remained at <1 ppm during 30 hours of continuous use of the cell. The oxygen removal system thus functioned as planned, and prevented contamination of NS-1 solvent by oxygen.

At the request of Commonwealth Edison Company, several known decontamination solvents were screened for their ability to initiate intergranular stress corrosion cracking (IGSCC) on highly sensitized 304 stainless steel U-bends. These solvents did not produce IGSCC under mild conditions, but neither did they remove heat-produced scale from 304 alloy specimens.

Under conditions of longer treatment times and higher temperatures such as those planned for use during the decontamination of Dresden-1, these other solvents initiated IGSCC similarly to the way in which Dow solvent

NS-1 initiated cracking (Table 3.3.7). Alkaline permanganate (AP) treatment followed by ammonium citrate (AC) or AC without the AP pretreatment did not appear to crack 304 stainless steel U-bends in 100 hours unless 0.12 wt/% Fe^{+3} was present. Specimens from several heats of 304 stainless steel sensitized 50 hours at 1200°F did not crack when exposed to this treatment at 250°F. When two double U-bends which had been exposed to AP followed by AC were placed in deionized water for 100 hours at 250°F, one exhibited cracking and the other did not.

Treatment with AP followed by CitroX solution (Table 3.3.7, (footnote) produced cracking on a specimen from one heat of 304 stainless steel, but did not produce cracking on a specimen from another heat of 304 steel in a 100 hour test at 250°F, with or without Fe^{+3} added.

Alkaline permanganate would be expected to degrade the GRAPHITAR 14 bearings in the reactor circulation pump of Dresden-1 since this material is known to degrade in the presence of alkaline oxidizing agents. (See Sections 3.3.3.6 and 3.4 for results with GRAPHITAR 14 in Dow solvent NS-1).

Phosphoric acid (60%) with inhibitor added did not produce cracking on highly sensitized 304 stainless steel in either a four hour or 100 hour test at 250°F. However, after 100 hours the specimens which were exposed four hours and then were rinsed and exposed to deionized water at 250°F for 100 hours did exhibit cracking.

In summary, when exposed to these known decontamination solvents, highly sensitized 304 stainless steel exhibited IGSCC similar to that produced by Dow solvent NS-1. Cracking is expected to occur on sensitized specimens which are not descaled or which are exposed to Fe^{+3} .

Table 3.3.7

Double U-Bend Stress Corrosion Data for 304 SS
Sensitized 50 Hrs at 1200°F, With Heat Treatment
Scale Intact, and Exposed to Known Decontamination Solvents

Solvent System	Treatment	Results ^g
APAC ^a	24 hours in AP at 250°F 28 hours in AC at 250°F	No cracking
Deionized H ₂ O	Same specimens as above exposed 100 hrs at 250°F	1 cracked, 1 did not
APAC	24 hours in AP at 250°F 100 hours in AC at 250°F	No cracking
AC ^b	100 hours at 250°F	No cracking
AC + 0.12 wt/%	100 hours at 250°F	No cracking
Inhibited 60% H ₃ PO ₄	2 hours at 250°F Repeat with fresh soln.	No cracking
Deionized H ₂ O	Same specimens as above exposed 100 hrs at 250°F	Cracking
Inhibited 60% H ₃ PO ₄	100 hours at 250°F	Severe crevice attack and metal loss
AP dilute ^c	100 hours at 250°F	No cracking
AP conc. ^d	100 hours at 250°F	No cracking
AP very dilute ^e	100 hours at 250°F	No cracking
AP Citrox ^f	2 hours in AP at 105°C, rinsed, 3 hours in Citrox at 80°C.	No cracking
AP Citrox	2 hours in AP at 105°C 100 hours in Citrox at 250°F	1 heat cracked 1 heat, no cracking
AP Citrox	24 hours in AP at 250°F 100 hours in Citrox at 250°F	1 heat cracked 1 heat, no cracking
Citrox	187 hours at 250°F	1 heat cracked 1 heat, no cracking
Citrox + 0.12 wt/%	187 hour at 250°F	1 heat cracked 1 heat, no cracking

Table 3.3.7 (Continued)

^aAPAC--AP: 100 g NaOH + 13 g KMnO₄/liter
AC: Diammonium Citrate - 13 g/liter

^bAC: Diammonium Citrate - 13 g/liter

^cAP-Dilute: 100 g NaOH + 13 g KMnO₄/liter

^dAP-Conc.: 200 g NaOH + 13 g KMnO₄/liter

^eAP-Very dilute: 6.5 g NaOH + 13 g KMnO₄/liter

^fAP CitroX: AP as AP-Dilute above
Citrox: Oxalic Acid - 25 g/liter
Diammonium Citrate - 50 g/liter
Ferric Sulfate - 2 g/liter
Diethylthiourea - 1 g/liter

^g(When cracking occurred it was IGSCC)

3.7 DISCUSSION

3.7.1 Sensitization and Stress Corrosion Cracking of Austenitic Stainless Steels

Much of the Dow solvent NS-1 corrosion study has been devoted to intergranular stress corrosion cracking of sensitized 304 stainless steel. Because even corrosion engineers do not completely agree on the theory and causes of such attack, an elementary discussion of the sensitization phenomena is in order for the reader who is unfamiliar with this subject.

The more common grades of austenitic stainless steels such as type 304 or 316 are limited by specification to 0.08 wt/% carbon. When used in the quench annealed condition (water quenched from 1900-2100°F) these steels retain the carbon in solid solution. However, when these steels are reheated to 850-1652°F, as occurs during welding or post weld heat treatment, chromium-rich carbides precipitate at the boundaries of the austenitic grains and the material is said to be sensitized. In other words, the carbon has exceeded its solubility limit for this temperature range.

The time-temperature corrosion susceptibility seems to reach a peak in the 1100-1200°F temperature range. Unfortunately, this peak falls into the 900-1150°F temperature range required by the ASME code for stress relieving of welded high strength steel components. Use of vessels or piping which contain both high strength steel and austenitic stainless steels in a manner such that both components must be stress relieved together will result in sensitization of the austenitic stainless steel components. Modern nuclear reactor design separates the austenitic stainless components from the high strength steel parts which require stress relief heat treatment after they are welded. In this case the 304 or 316 stainless steel subject to sensitization or carbide precipitation only from the heat of welding.

The grades of stainless steel which have extra low carbon contents, such as types 304L or 316L in which the carbon content is limited to 0.03% maximum, do not exceed the solubility limit for carbon if they are not held at sensitization temperatures for prolonged periods. These materials are not susceptible to sensitization while they are being welded but can become partially sensitized and subject to intergranular corrosion in some media if subjected to prolonged post weld heat treatment (PWHT).

Because carbon has a greater affinity for Niobium (columbium) or tantalum than it does for chromium, stabilized grades of stainless steel such as type 347 or 321 which contain these elements are sometimes used where resistance to sensitization is desirable. In these stabilized grades of stainless steel the intermetallic carbides of Niobium (Columbium) and tantalum are dispersed throughout the austenite grain region or more often are concentrated discontinuously at the grain boundary. Like the extra low carbon grades of austenitic stainless steels, these stabilized alloys resist sensitization in the relatively short periods during which they are exposed sensitization temperatures under normal welding operations. Prolonged heating at sensitization temperatures either during PWHT or welding of the alloys still can leave them susceptible to a form of intergranular corrosion known as knife-line attack in some media.

Possibly, different mechanisms are involved in different cases of intergranular corrosion, depending on the chemical composition and metallurgy of the particular heat of stainless steel involved and on the nature of the corroding media. However it is not axiomatic that intergranular corrosion will occur in the presence of a continuous grain boundary precipitate of heavy carbides. Many otherwise fairly corrosive media do not cause intergranular corrosion on sensitized stainless steels although these media may result in significant general corrosion and weight loss.

Transgranular stress corrosion cracking of austenitic stainless steel is common, particularly in media which contain high concentrations of chlorides at elevated temperatures and in the presence of high stress levels in the base alloy. Such transgranular stress corrosion cracking occurs regardless of the degree to which the stainless alloy is sensitized, but seldom is noted when the steel is undergoing general corrosion. Conversely, intergranular stress corrosion cracking of austenitic stainless steels is relatively rare. Most examples reported in the literature for nuclear reactors are associated with those exposed to high temperatures or high purity water of intermediate oxygen content. Although large areas of the exposed stainless surface may be heavily sensitized, attack seems limited to very small areas of the surface which have been subjected to high levels of cold work.

Generally it is recognized that stress corrosion cracking occurs in several stages: a long initial period for crack initiation followed by a relatively short period for crack propagation. Cathodic protection is known to prevent initial cracking or halt propagation of stress corrosion cracking.

It is particularly difficult to predict from laboratory studies whether or not intergranular or transgranular stress corrosion cracking will occur in actual service. Duplicating all of the variables (metal composition, thickness, metallurgical history, stress level and type, etc.) of the operating equipment on a laboratory basis is almost impossible with small samples of stainless steel. Hence, the tendency in laboratory studies is to attempt to obtain corrosion or cracking in a short period of time by exaggerating test variables such as temperature, stress level, degree of sensitization, contaminant levels, etc.

Some specimens crack while others do not under the same conditions of exposure. Even if large numbers of identical samples are evaluated to obtain statistically significant distribution curves for failure probability these data can not be extrapolated to actual operating equipment experience with certainty because of the large number of known and unknown variables which can affect cracking susceptibility.

Corrosion engineers recognize that the following factors are only a few of those that can influence stress corrosion cracking trial results.

- . Material variation
 - 304 SS
 - 316 SS
 - etc.
- . Compositional variation of a material from heat to heat
- . Metallurgical history variation of a material from heat to heat
- . Type of specimen, dimensions and geometry
 - U-bends
 - Bent beams
 - Pressurized tubes
 - Linear elastic fracture mechanic specimens
 - Uniaxial tensile specimens
- . Stress level and type
- . Degree of sensitization (time and temperature)
- . Stressed before or after sensitization
- . Cold work level and type

- . Metallographic characteristics
 - Inclusions
 - Other phases
 - Heterogeneties
 - etc.
- . Surface finish
 - Roughness
 - Ground or polished (direction)
 - Machined or milled
 - Pickled (specific pickling agents)
 - Grit blasted (with sharp or rounded particles)
 - Tensile or compressive stresses
- . Orientation effects (samples cut at right angles to direction of rolling will perform differently from those cut parallel to direction of rolling)
- . Form
 - Bar
 - Rod
 - Sheet
 - Strip
 - Plate
 - Rolled
 - Forged
 - Cast
- . Stress concentration factors
- . Surface film characteristics
 - Thickness
 - Composition
 - Strain (kinetics)
 - Dislocations
 - Crystallographic structure
- . Environment
 - Time
 - Temperature
 - Pressure
 - Oxygen concentration
 - Inhibitors (natural or added)
 - Accelerators (natural or added)
 - Chlorides, fluorides
 - Other ions and compounds
- . Strain
 - Magnitude
 - When applied (before or after sensitization, in plastic or as permanent deformation region).

In addition to those factors already known to influence stress corrosion cracking, probably many more exist which have not been recognized to date.

The absolute values of the above factors in operating equipment. cannot be determined. Duplicating these exact conditions in laboratory trials cannot be carried out with certainty. Little agreement exists among corrosion authorities on the exact mechanics of stress corrosion cracking (intergranular or transgranular) initiation and propagation or on methods to eliminate or control this form of corrosive attack.

Stress corrosion cracking studies designed to simulate the operating environment of actual equipment can be very sophisticated and extensive. Even with a wealth of data from such tests, the materials engineer often finds it difficult to judge whether or not stress corrosion cracking will occur during actual operating conditions.

3.7.2 Loop Test on Irradiated Stressed Samples 304 SS From Dresden-1

Four highly irradiated Charpy containers of 304 stainless steel were removed from the Dresden-1 reactor vessel and were used to fabricate 26 tensile dog-bone coupons. These specimens will be tested under simulated cleaning conditions in a dynamic test loop. Constant stress tensile jigs have been fabricated as shown in Figure 3.7.2. A dynamic loop will be built to carry out these tests. The results will be disseminated in a separate report.

Figure 3.7.2
Constant stress jig for testing
irradiated 304 SS.

CONCLUSIONS

The following conclusions were drawn from this comprehensive study of the corrosion effects of Dow solvent NS-1 on the materials of construction used in the Dresden-1 boiling water reactor.

1. No significant corrosion problems are anticipated on any of the alloys at Dresden-1 when they are cleaned with Dow solvent NS-1 at the planned decontamination process temperature.
2. Electrochemical tests and artificial crevice tests indicated that Dow solvent NS-1 does not cause crevice attack on the stainless alloys and hard-facing alloys.
3. Stressed crevices of sensitized 304 stainless steel filled with Dow solvent NS-1 saturated with Fe_3O_4 showed no evidence of attack after 100 hours at 250°F followed by 200 hours at 500°F in deionized water.
4. Evaluation of wedge open loaded (WOL) precracked specimens of sensitized 304 stainless steel exposed to Dow solvent NS-1 and deionized water indicated that NS-1 solvent is no more aggressive than deionized water on a stressed crevice.
5. Tests of Dow solvent NS-1 sequestered in an artificial crevice of sensitized 304 stainless steel indicated no crevice attack occurred. Heating these crevices with the entrapped solvent to 500°F in Deionized water produced no adverse attack from solvent decomposition products.
6. A 10-day dynamic loop test of GRAPHITAR bearings in a small immersion pump, similar in design to the main coolant pumps in Dresden-1, indicated that the GRAPHITAR bearing material is inert to Dow solvent NS-1.
7. Up to nine times the normal concentration of Dow solvent NS-1 was intermittently in contact with 304 stainless steel at 250°F for six months with no adverse effects.
8. Welds of 304 stainless steel up to 5/8" thick were exposed to Dow solvent NS-1 with no adverse effects.
9. The concentration of the corrosion inhibitor in Dow solvent NS-1 should be twice the standard use level to keep general corrosion rates of the 300 series stainless and carbon steel alloys at the lowest possible levels.

10. Impurities harmful to sensitized and stressed 304 valve packing, FLEXITALLIC gasketing, and asbestos gasketing were these materials exposed to Dow solvent NS-1.
11. No intergranular stress corrosion cracking was found on any 304 stainless steel specimens which had been sensitized and stressed beyond their yield valves after they are exposed to Dow solvent NS-1 at times and temperatures exceeding those anticipated in the proposed cleaning program.
12. IGSCC was initiated on 304 stainless steel U-bends under accelerated condition which included:
 - a. exaggerated sensitization and heavy carbide precipitation by exposure of at 1200°F for at least 30 hours prior to stressing them into U-bends.
 - b. The presence of heat treatment scale as a result of sensitization of the specimens in air for prolonged periods. However, if the scale was removed, the specimens did not display IGSCC unless subjected to the condition noted in paragraph "c" below.
 - c. The presence of high concentrations (>0.12 wt%) of ferric ion in the presence or absence of nickelous ions and hexavalent chromium ion (0.1%).

Note: Condition "a" coupled with "b" and/or "c" can cause stress cracking of specimens exposed to Dow solvent NS-1. However, these same conditions cause stress cracking of specimens exposed to deionized water. Hence, it is concluded that conditions of such exaggerated severity do not exist at Dresden-1 since any reactor construction materials exposed to such conditions would have long since failed.

Appreciation is expressed to Mr. W. P. Worden of the Commonwealth Edison Company for his support of the corrosion study program. His insistence on a thorough investigation of every form of corrosion and every material which could result in a safety hazard at Dresden-1 ensured the completion of comprehensive corrosion trials prior to the actual decontamination. Valuable contributions to the corrosion study program were also provided by W. Kiedaisch, S. Gaitonde and R. Tammingo also of the Commonwealth Edison Company.

The significant contributions of Dr. C. F. Cheng of Argonne National Laboratories and Dr. R. W. Staehle of the Ohio State University, consultants to Commonwealth Edison Company, and W. L. Walker, T. E. Adams and others in the General Electric Company, Nuclear Division in the development of the corrosion study program and in the interpretation of results are gratefully acknowledged.

Members of the Contract Research Department Staff, Texas Division, Dow Chemical U.S.A. whose work on the program should be recognized include O. Osborn, C. F. Schrieber, J. A. Manning, S. N. Smith, E. F. Sablatura, G. R. Bullard and F. Garcia.

3.10 General Electric Corrosion Test Procedures

The Commonwealth Edison Company requested that experts from General Electric Company review the corrosion data generated by Dow Nuclear Services and to generate the necessary backup data to ensure that all questions relating to the corrosivity of Dow solvent NS-1 had been fully studied.

Three tasks developed from a screening of the corrosion data obtained at Dow:

1. Evaluation of stress corrosion cracking (SCC) of sensitized type 304 stainless steel during simulated cleaning cycles, and confirmatory tests of stress corrosion data from Dow relating to the effects of iron dissolved in the solvent.
2. Evaluation of the effect of exposure of sensitized 304 stainless steel to Dow solvent NS-1 on the subsequent in-service SCC behavior of the alloy.
3. Provision of samples for surveillance testing during the actual Dresden-1 cleaning operation, and for continued monitoring of subsequent SCC behavior of test samples exposed in the system during the cleaning operation.

Task 1 Summary

Three runs were made under Task 1.

The first run was designed to demonstrate that the occasional SCC of sensitized 304 stainless steel U-bends observed in 250°F deionized water was the result of the severely cold-worked bend areas of the samples, and not a characteristic of sensitized 304 stainless steel in general. The heat (mo. 78500) of 304 stainless steel used in this and all other runs was selected on the basis of its demonstrated low resistance to intergranular SCC in high temperature, high purity oxygenated water.

Ten rectangular bent beam samples were heat treated in air at 1200°F and were cooled in the furnace. Heat-produced oxide was removed from all surfaces of five samples by using a wet belt power grinder, with 120 grit. The remaining five samples were tested with heat-produced oxides intact. The samples were loaded on constant radius blocks to give 2% strain in the outer fibers, and were exposed to air-saturated deionized water at 250°F for 100 hours under static conditions. The samples then were examined optically at magnifications of up to 30 diameters while still loaded on the blocks. In addition, examinations of the stressed surfaces of the samples treated with liquid penetrant were made with the samples still loaded in tension. The liquid penetrant tests on the surfaces revealed a few small indications but the optical examination revealed that the indications were small surface defrets and did not represent SCC.

The second run under this task was designed to evaluate the SCC effects of NS-1 solvent on sensitized 304 stainless steel. The run was conducted in the same manner as the first run, except that fresh Dow solvent NS-1 was used, and oxides were removed from five of the ten samples by dry blasting them with 240 grit.

Optical and liquid penetrant examinations of the loaded samples after completion of the run, showed no indications of SCC.

The third run was designed to evaluate the effects of expected contaminants in NS-1 solvent. This was conducted in the same manner as the second run, except that the specimens were exposed to simulated used NS-1 solvent which contained 0.12 weight% of Fe^{+3} and 0.65 weight % of Ni^{+2} . Optical and liquid penetrant examinations of the specimens after completion of the run showed not indications of SCC.

Task 2 Summary

The Task 2 tests were performed to evaluate the effects of NS-1 solvent on the subsequent SCC behavior of sensitized 304 stainless steel in high purity 550°F water containing 0.2 ppm oxygen. Work performed by the General Electric Boiling Water Reactor Systems Department in earlier investigations showed that certain acid solutions produced what has been termed a "pickling effect"

whereby short exposures to the solution significantly increased the probability of SCC in subsequent exposure to high temperature oxygenated water. This effect is not a function of the degree to which the solution produces intergranular attack during the pre-exposure, but rather appears to be a surface effect which is specific to the composition of the solution itself. Furthermore, the pickling effect was observed only at low stress levels in the tests on fixed deflection, bent beam samples where relaxation occurred. Identically treated uniaxial tensile samples exposed at 125% of the 550°F, 0.2% yield strength did not exhibit significant differences in SCC behavior.

Two types of samples were prepared for the Task 2 tests: cylindrical gage section, uniaxial tensile samples, with gage diameters to give stresses of 75%, 100%, and 125% of the 550°F, 0.2% yield strength; and rectangular fixed deflection bent beam samples. All samples were sensitized by heat treating them for 50 hours at 1200°F, after which they were allowed to cool in the furnace. Heat-produced oxide was removed from all surfaces of the samples by blasting them with dry 240 grit silicon carbide. Thirty tensile samples and 12 bent beam samples were prepared. Half of each group of samples was set aside as controls, and the other half of each group was exposed in the unstressed condition to simulated used NS-1 solvent at 250°F for 100 hours. Following the exposure the samples were rinsed with demineralized water and then were loaded into specimen holders along with the unexposed control samples.

Time-to-failure of the tensile samples is signaled by a pressure surge which occurs when the loading piston is forced rapidly into a manifold. This signal is then recorded on a continuous strip chart. The failed sample is identified by a number on the failure indicator ball which falls into the collector tube at the bottom of the manifold. The only means of determining whether or not stress corrosion cracking has occurred on the bent beam samples is by periodic optical examination following shutdown of the autoclave and removal of the samples.

Total exposure time accumulated to date is approximately 9000 hours. Bent beam samples examined at approximately 600, 1400, 2800 and 5300 hours gave no indications of cracking. No failures have occurred in the uniaxial tensile samples. The bent beam samples will be examined next at approximately 10,000 hours total exposure.

Task 3 Summary

The tests to be performed under this task represent evaluations of the SCC effects of Dow solvent NS-1 during actual cleaning operations, and the effects of the cleaning operation on subsequent long-term SCC behavior of samples in actual operating reactor coolant. The preliminary test program involved a limited number of samples exposed during the cleaning of the General Electric

Corrosion Test Loop at Dresden-1. Results of the preliminary test program appear in a separate report. The full program involves large numbers of samples of a variety of materials which will be exposed during cleaning of the Dresden-1 primary system. The samples consist of tubes in which stresses are introduced by internal pressurization. The final stress levels are controlled by pressurizing the tubes to an initial precalculated value with the final pressure reached as the gas comes into thermal equilibrium with the reactor coolant at operating temperatures. Since SCC on these samples can only be determined visually, periodic examinations are required.

Pressurized tube samples were prepared from both as-received 304 stainless steel (heat no. 78500) and as-welded 304 stainless steel (heat no. 78500), where the weld was approximately in the center of the tubular section. After the as-received samples were fabricated they were sensitized for 50 hours at 1200°F and allowed to cool in the furnace. The samples then were pressurized to give a hoop stress of 100% of the 550°F, 0.2% yield strength (17.6 Ksi) at reactor operating temperatures. Fourteen welded samples and 12 sensitized samples were prepared.

The samples were loaded into two capsule holders, with seven welded and six sensitized samples in each holder. One sample holder was placed in the General Electric Corrosion Test Loop at Dresden-1 during the cleaning of that system while the other was held as a control. The samples were then to be exposed in the test loop to the reactor coolant. Results of these tests are contained in a separate report.

Fabrication of samples for the full surveillance program will begin in 1977.

The summary of the General Electric tasks and results to date were taken from the first progress report entitled "Dresden-1 Radiation Level Reduction Program Corrosion Tests."