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RADIATION LEVEL PROGRAM CORROSION TESTS

PROGRESS REPORT

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NUCLEAR ENERGY DIVISION

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PLANT MATERIALS ENGINEERING
INTERIM REPORT

LONG TERM EVALUATION OF THE EFFECTS OF EXPOSURE TO DOW NS-1 SOLVENT ON THE STRESS CORROSION CRACKING OF SENSITIZED TYPE 304 STAINLESS STEEL IN SIMULATED BWR ENVIRONMENT

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INTRODUCTION

Past work conducted by General Electric Company's Nuclear Energy Engineering Division (NEED) in San Jose, CA, has produced evidence that, under certain circumstances, short term exposures to specific corrodants at moderate temperatures can have very significant effects on the intergranular stress corrosion cracking (IGSCC) behavior of sensitized Type 304 stainless steel when subsequently exposed in the stressed condition to oxygenated high temperature high purity water. The corrodants which have produced this effect have been strong inorganic acids with significant quantities of hydrofluoric acid present. Exposure to strong inorganic acids without the presence of hydrofluoric acid has not resulted in enhanced susceptibility to IGSCC in the tests performed to date, and the general effect has been called the "pickling effect" because of the apparent association with the fluoride ion. However, extensive testing to determine the mechanism of the pickling effect and to clearly demonstrate that it is specifically associated with the fluoride ion has not been performed. In the absence of such definitive testing, it has been necessary to adopt the conservative position that any acid solution might be capable of producing the pickling effect, and to prohibit the exposure of austenitic stainless steels to acidic solutions, except in the solution annealed condition. This position has been implemented in NEED purchase and installation specifications for several years.

The composition of the proposed solvent (NS-1) for the Dresden 1 primary system cleaning operation is proprietary to the Dow Chemical Company, however, NEED has been informed that it does contain one or more organic chelating and complexing agents, and has a pH of 3 to 4. In the absence of information to the contrary, it was believed that tests to evaluate the presence of a possible pickling effect from this solution were necessary in order to assure that no serious deleterious effect resulted from exposure to NS-1. This report presents the results of the investigation into this question.

SUMMARY

Multiple specimens of furnace sensitized Type 304 stainless steel were exposed to a simulated cleaning cycle in "used" Dow NS-1 solvent, and then to 13,503 hours in a simulated BWR environment; 0.2 ppm oxygen, 288°C (550°F) high purity

water. Five specimens were exposed to NS-1 and then strained 2% as bent beams during the subsequent BWR exposure, and six specimens each were exposed to NS-1 in the unstressed condition and then exposed to the BWR environment as uniaxial tensile specimens at stresses of 75%, 100%, and 125% of the 288°C (550°F) 0.2% offset yield strength. Duplicate sets of control specimens were exposed to 121°C (250°F) demineralized water, and then to the BWR environment.

Interim optical examinations were made during the simulated BWR exposure, and final scanning electron microscope and destructive metallographic examinations were made following the 13,503 hours exposure. No indications of stress corrosion cracking were observed on any specimen. The data generated in this investigation indicate that exposure to Dow NS-1 does not produce a significant increase in the susceptibility of sensitized Type 304 stainless steel to intergranular stress corrosion cracking in subsequent BWR service. Some shallow intergranular penetrations were observed on the bent beam specimens, but these penetrations were shown to be original intergranular attack on the inside diameter surface of the pipe which occurred during its manufacture.

DETAILED DISCUSSION

Material

The heat of material chosen for this investigation (Heat No. 78500) was selected on the basis of its susceptibility to intergranular stress corrosion cracking (IGSCC) in constant load tests performed at higher stress levels in oxygen-saturated high purity water at 288°C(550°F)[1], and the ability to machine all required specimen configurations from the as-received material. The original configuration of the material was 15 cm (6 in) Schedule 80 pipe, with the composition and mechanical properties shown in Tables 1 and 2 respectively. The as-received microstructure of this heat is shown in Figure 1.

All specimens were tested in the furnace sensitized condition - 50 hours at 648°C (1200°F), furnace cool - and the response of this material to an ASTM A 262, Practice A, test after heat treating is shown in Figure 2.

Specimen Configuration

Two specimen types were used in this investigation; fixed deflection bent beams and uniaxial tensiles, shown in Figures 3 and 4 respectively.

Six bent beam specimens were exposed to NS-1 prior to the simulated BWR exposure and six specimens were exposed to 121°C (250°F) demineralized water to act as controls; all in the unstressed condition. The bent beam specimens were then stressed by bending them over solid fixed radius blocks, with a radius chosen to produce 2% strain in the outer fibers of the specimens, as shown in Figure 5, prior to the long term simulated BWR exposure.

The uniaxial tensile specimens were not stressed during the NS-1 and demineralized water control exposures, but were exposed to simulated BWR service environment as constant load specimens stressed to 75%, 100%, and 125% of the 288°C (550°F) 0.2% offset yield strength of the material. The specimens were loaded into the test modules shown in Figure 6 and stressed by internal autoclave pressure, with the diameter of the reduced gage sections adjusted to give the selected stress levels on the specimens. Five specimens for each stress level were exposed to NS-1 prior to the simulated BWR exposure, and five specimens were exposed to 121°C (250°F) demineralized water to act as controls.

Specimen Exposure

The solution used to simulate the cleaning cycle was Dow NS-1 with additions of 10.4 grams/liter ferric ammonium sulfate [$Fe(NH_4)(SO_4)_2 \cdot 12\ H_2O$] and 2.9 grams/liter nickel sulfate ($NiSO_4 \cdot 6\ H_2O$) to simulate the solvent at the maximum allowable iron and nickel concentrations of 1200 ppm and 650 ppm, respectively, specified for the cleaning operation ("used" NS-1). The initial exposure of specimens was to a cycle of approximately 100 hours at a temperature of 121°C $\pm$ 5°C (250°F $\pm$ 10°F), with one set being exposed to "used" NS-1 and the control set being exposed to demineralized water.

All 121°C (250°F) specimen exposures were performed in a TFE Fluorocarbon-lined pipe spool piece, with TFF-lined blind flanges, to avoid contamination of the solution with extraneous material. Solution volume-to-surface area ratios were approximately 40 l/m^2(1 gal/ft^2) during both exposures, and both solutions were essentially air-saturated at the time of initial immersion of the specimens. No attempts were made to remove oxygen from the solutions prior to insertion of the specimens because, at that time, the intent was that no degassing operation would be performed in conjunction with the actual plant cleaning operation. This position was subsequently changed and a nitrogen sparging and blanketing step was added to the plant cleaning procedure. The variance of the test procedure from that eventually planned for the plant cleaning did not represent a significant difference from the standpoint of the purpose

of the test because, if anything, the presence of oxygen would tend to increase the probability of the occurrence of intergranular corrosion of sensitized Type 304 stainless steel.

Following the NS-1 and demineralized water control exposures, the specimens which had seen NS-1 were rinsed lightly with demineralized water to remove residual solvent. The bent beams were paired on their radius blocks and plastically deformed by bolt loading, and the uniaxial tensile specimens were loaded into their test modules. All specimens were then exposed to simulated BWR service in water containing 0.2 $\pm$ 0.1 ppm dissolved oxygen, with a conductivity of less than 1.5 μ mhos, at a temperature of 288°C $\pm$ 5°C (550°F $\pm$ 10°F). Flow rate through the autoclave was 50-60 cm^3/min, and conductivity was continuously monitored. Daily measurements of dissolved oxygen and pH were made and recorded. Total exposure time was 13,503 hours, except for single bent beam specimens from each group which were removed for metallographic examination at 11,700 hours for purposes of another investigation[2].

Specimen Examinations

Periodic examination of the specimens was not a planned part of the program. The intent was to perform examinations whenever a constant load uniaxial specimen signaled a failure. However, only one such event took place at approximately 600 hours exposure, and that event was a false signal of failure. The bent beam specimens were examined optically at magnifications up to 30 diameters after the false failure signal at 600 hours exposure, and during an autoclave repair outage at 12,924 hours exposure. No interim examinations of the uniaxial tensile specimens were made because of the difficulty in removal and replacement in the test fixture, and their capability of signal of failure. As mentioned previously, a single bent beam specimen from each of the NS-1 and control sets was removed and subjected to destructive metallographic examination after 11,700 hours exposure to confirm the absence of general intergranular corrosion resulting from the NS-1 exposure. An additional 579 hours exposure was accumulated, for a total of 13,503 hours, prior to an extended test facility outage for instrument calibration and system modification. At that time, the tests were terminated.

Following the 13,503 hours exposure, all specimens were examined optically at magnifications up to 30 diameters, with the bent beam specimens still stressed on their radius blocks. Single specimens from both the control and NS-1 groups were then examined by scanning electron microscopy (SEM) at magnifications up to 1,500 diameters. Finally, destructive metallographic examinations were performed

on specimens subjected to SEM examination.

RESULTS

As indicated previously, the only indication of stress corrosion cracking failure of a specimen during the 13,503 hour simulated BWR exposure proved to be a false indication. No failures of any type were observed during the course of the long term exposure.

Optical examinations of the specimens following completion of the BWR exposure gave no indications of the presence of stress corrosion cracking. However, several bent beam specimens from both the control group and the NS-1 group exhibited areas of oxide which appeared to reflect the underlying grain structure of the base metal. Photographic resolution of these areas did not prove possible because of the subtle contrast differences which were visually apparent. There did not appear to be any significant difference in this respect between the control specimens and the NS-1 specimens, and the effect was classified simply as a testing artifact.

The as-tested surfaces of single control specimens and specimens which had been exposed to NS-1 prior to the long term BWR exposure from each test group were subjected to scanning electron microscope (SEM) examination. All specimens exhibited normal high temperature water corrosion product films, and no indications of intergranular stress corrosion cracking were observed. Typical surface conditions are shown in Figures 7 through 14.

Following the SEM examinations, the specimens were sectioned at their approximate midplanes and longitudinal sections were mounted and polished for each specimen. The uniaxial tensile specimens exposed at 75%, 100%, and 125% of the 288°C (550°F) 0.2% offset yield strength all exhibited normal surface conditions with continuous oxide films and no indications of intergranular stress corrosion cracking, as shown in Figures 15 through 17, respectively. Similarly, no indications of intergranular stress corrosion cracking were observed on the demineralized water control bent beam specimen, as shown in Figure 18, However, a number of shallow intergranular penetrations were observed on the bent beam specimen which had been exposed to NS-1 prior to the simulated BWR exposure, as also shown in Figure 18.

The intergranular penetrations observed on the NS-1 bent beam specimen differ from typical intergranular stress corrosion cracking penetrations in a number of

ways. First, the penetrations are filled with oxide which is continuous with the surface oxide film, as shown in Figure 19, and this accounts for the lack of their observance during the SEM examination. Intergranular stress corrosion cracks have invariably shown discontinuous corrosion product films at the surface, and a discontinuous film is a requirement for stress corrosion cracking in the currently accepted models because there must be a path to the crack tip which permits access of the corrosive fluid. Second, the tips of the intergranular penetrations are blunted while intergranular stress corrosion cracks have consistently shown sharp crack tips. Finally, if one assumes that the initial penetrations were the result of intergranular stress corros cracking, then one must conclude that the propagating cracks arrested for some reason and that general corrosion of the exposed crack faces resulted in the blunting of the crack tip and the filling of the crack with corrosion product. To the best of this writer's knowledge, this situation has not been observed in any of the service failures or laboratory test failures of sensitized Type 304 stainless steel. None of the characteristics of the penetrations observed on the bent beam specimen are consistent with those associated with intergranular stress corrosion cracking, except for the intergranular nature of the penetrations themselves. The evidence would indicate that the intergranular penetrations were present on the original specimen surface prior to exposure to the simulated BWR environment, and may have been present prior to the exposure to the NS-1 solution.

The most probable source of intergranular penetrations on stainless steel piping is the pickling operation at the manufacturer's shop, and numerous instances of such intergranular attack have been observed in the past. The attack occurs as a result of either an improperly controlled pickling bath, or surface carburization of the pipe during some step in its manufacture. The inside diameter surface of the pipe from which the specimens for this program were fabricated was examined, and intergranular attack was observed in the as-received condition, as shown in Figure 20. However, the bent beam specimens were machined on all surfaces prior to testing in order to produce a uniform starting surface condition. If the observed intergranular penetrations on the test specimen were caused by pickling attack during manufacture and insufficient material was removed by the machining operation, then one would expect them to exhibit certain characteristics. First, they should be relatively blunt penetrations because of the very agressive nature of the pickling solution. Second, they should be filled with a continuous corrosion product film after long term simulated BWR service

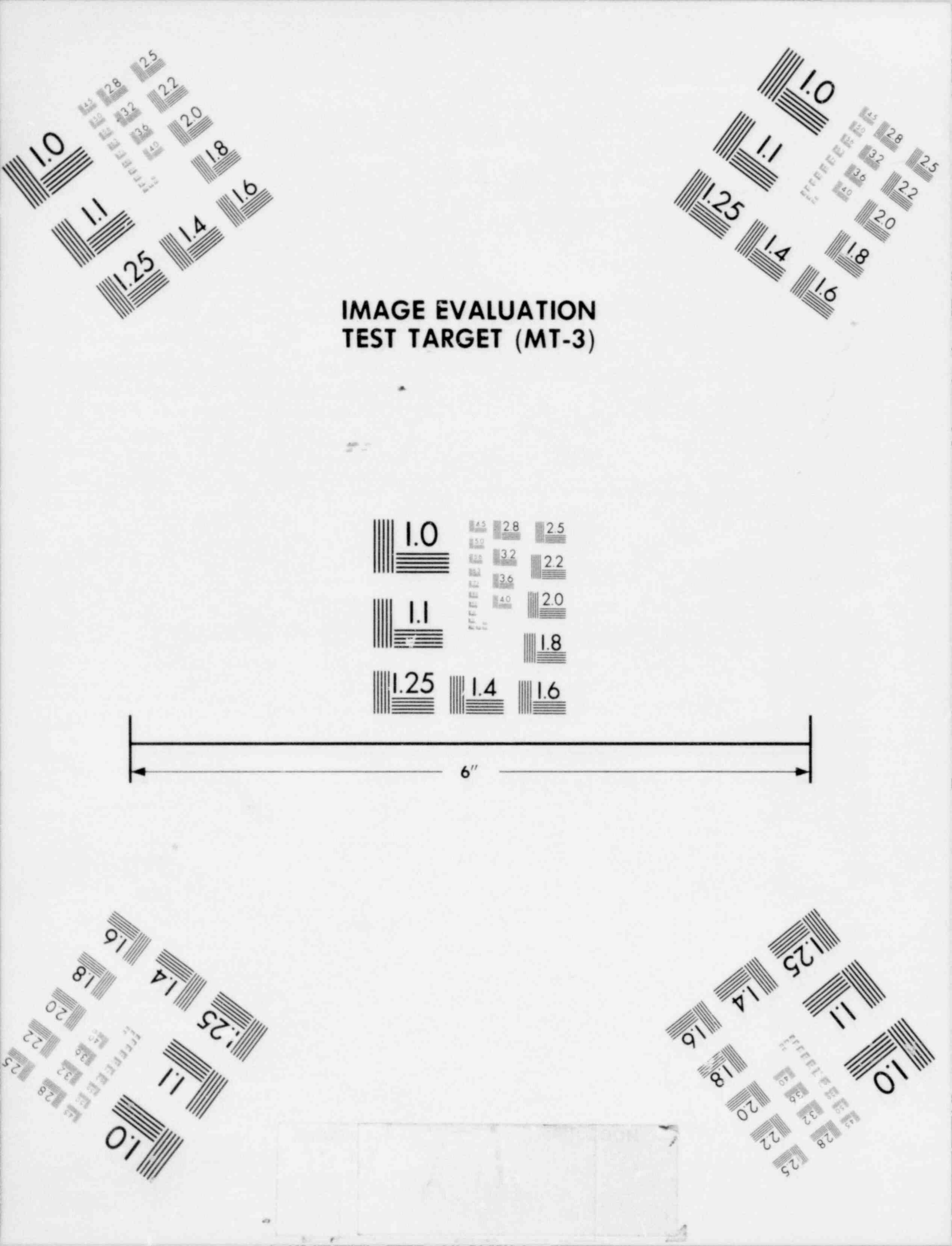
IMAGE EVALUATION
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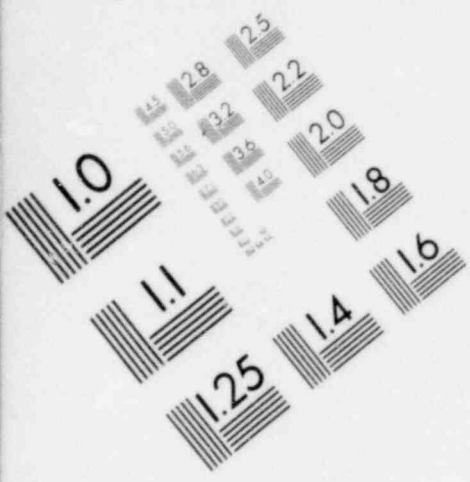


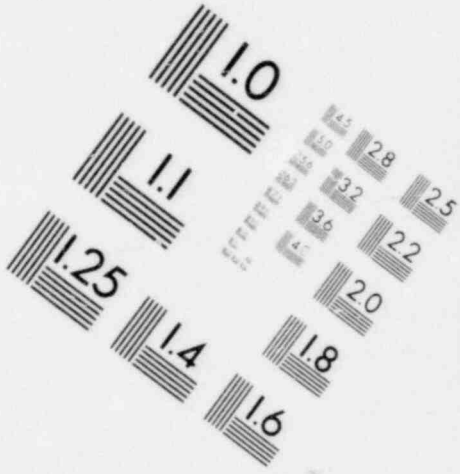
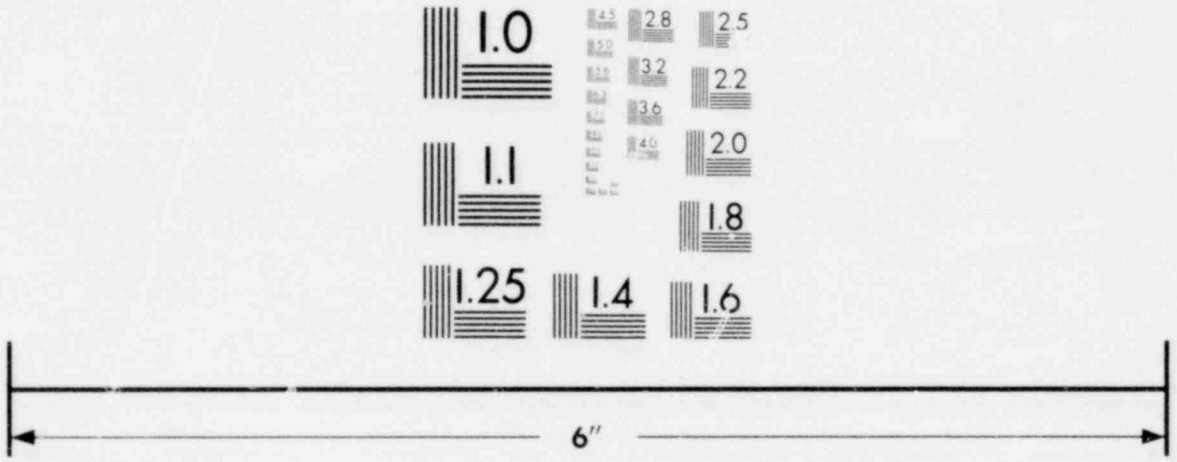
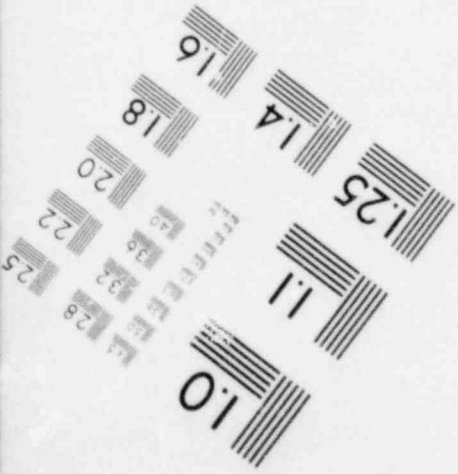
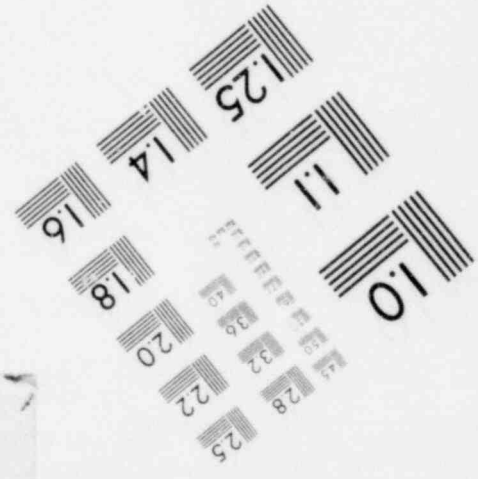


IMAGE EVALUATION TEST TARGET (MT-3)







exposure because the side walls of the penetration would corrode at the same rate as the general surface of the metal, and there would be no driving mechanism for further separation of the relatively close walls of the original penetration. Third, the density of penetrations should change in a predictable manner as one examined different sections through the same specimen, and the observance of such penetrations should be a sporadic occurrence on only the bent beam specimens because of the manner in which the specimens in this test were machined and tested.

The reduced gage section of the uniaxial tensile specimens would be located at the approximate mid-wall region of the pipe, as shown in Figure 21. Machining in this manner would have removed all traces of any original intergranular attack from the finished specimen because no tested surface would have been near the original inside diameter pipe surface. One would not expect to observe any intergranular penetrations on the tensile specimens which was the result of intergranular attack during the fabrication of the pipe. However, the machining of the bent beam specimens does present the possibility of the retention of original manufacturing intergranular attack on one surface of the finished specimen. As shown in Figure 22, a bent beam specimen can be machined from one of three general locations; near the outside diameter of the original pipe, in the mid-wall area, and near the inside diameter of the pipe. The situation which would produce a specimen with intergranular penetrations on an as-machined surface would be one in which the specimen was machined from near the inside diameter surface, as shown in Figure 22-C, and the manner in which the bent beam specimens were machined would tend to maximize the probability of one surface being near the original pipe inside diameter surface.

The specimens were machined in the following sequence of steps. First, a longitudinal section of approximately the length and width of the finished specimen would be saw-cut from the pipe. Second, the rough blank would be placed on the milling table with the inside diameter surface down, and one major face would be machined flat. The amount of material removed from the outside diameter surface would depend upon the degree to which the blank sat "flat" on the table. After machining the outside diameter surface, the specimen would be turned over and the inside diameter surface would be machined to the final thickness. Finally, the sides of the specimen would be machined to the final dimension. The amount of material removed from the inside diameter surface would depend upon how much had been removed from the outside diameter surface. The probability of any original surface remaining on the outside diameter specimen face would be

near zero, because of the curvature of the pipe surface. However, the probability of some original surface remaining on the inside diameter specimen face would appear to be significant, but of unknown magnitude because of the interaction of the two variables of the orginal specimen blank geometry and the amount of material which was removed from the outside diameter surface during the first machining operation.

Furthermore, even if every bent beam specimen had some vestige of original intergranular attack on the inside diameter specimen face, the probability that the inside diameter face was stressed in tension would be only 0.5 because no identification of original specimen orientation was maintained. The combination of the uncertainties related to the physical location of the specimens within the original pipe wall, and the particular specimen face stressed in tension, makes destructive examination of all specimens a questionable procedure for the resolution of the source of the observed intergranular penetrations.

A definitive method for demonstrating that the source of the intergranular penetrations was original pickling attack at the mill involves the distribution of the penetrations on the specimen. If the penetrations represent original intergranular attack, then a maximum density of penetrations should be observed at some point on the specimen and should decrease to zero toward the edge of the specimen. This would result from the concave nature of the inside diameter surface, with more material being removed from both edges of the specimen than from the center during machining. Stepwise polishing and etching through the specimen width would provide the necessary information.

The original mounting instruction for this specimen called for sectioning through the approximate mid-width plane, and the precise location of the sectioning cut was not recorded. In addition, the amount of material removed during the initial polishing operation was not measured, so it was not possible to ascertain the precise location of the plane examined or the amount of remaining material. A single incremental step grind of 0.13 cm (0.05 in) was made and the specimen was repolished and examined. No change in density of intergranular penetrations was observed, but there was also no significant change in the maximum depth of 1×10^{-3} cm (0.0004 in). Furthermore, there were no indications that the intergranular penetrations observed were continuous through the distance removed; a characteristic which one would expect if the penetrations represented intergranular stress corrosion cracking.

It was considered desirable to retain as many of the specimens as possible in their as-tested condition until the question of the source of the intergranular penetrations was resolved. For this reason, no additional specimens were mounted for sequential sectioning and polishing. Instead, the two specimens which had been removed after 11,700 hours exposure were used for further investigation. These two specimens, one control specimen and one NS-1 specimen had been mounted on an original side face and polished, with only a small amount of material removed from the full width of the original specimens. The specimens were ground and polished in three sequential 0.13 cm (0.05 in) increments, and the number of intergranular penetrations and their locations were recorded for each section examined. The results are shown in Table 3.

The data shown in Table 3 exhibit the types of patterns which would be expected to occur if the intergranular penetrations observed were the result of original intergranular attack of the pipe inside diameter surface during its manufacture. The density of penetrations changes from a minimum to a maximum as one examines successive sections across the width of the specimen.

In addition, there were no indications that any of the penetrations observed were continuous over the distance moved across the specimen width. This would appear to be conclusive evidence that the observed penetrations were caused by a pickling operation during the manufacture of the pipe.

CONCLUSION

The results of this investigation do not indicate the presence of a significant deleterious effect of exposure to Dow NS-1 on the stress corrosion cracking behavior of sensitized Type 304 stainless steel in subsequent exposure to a simulated BWR environment over long periods of time. One cannot make a statistical statement as to the confidence associated with this conclusion because no failures occurred in either the NS-1 group or the control group. However, the tests were run under conditions in which the presence of such an effect has been observed in previous studies performed by General Electric Company's Nuclear Energy Engineering Division.[3]

REFERENCES

1. Walker, W. L., Correlation Analyses of Mechanical Properties, General Intergranular Corrosion, and Intergranular Stress Corrosion Cracking Data from Multiple Heats of Type 304 Stainless Steel, NEDM-13347, Class I, July, 1973.

3. Walker, W. L., Investigation of the Effects of Exposure to Dow NS-1 Cleaning Solvent on Intergranular Corrosion of Sensitized Type 304 Stainless Steels, PME Transmittal No. 78-509-010, January, 1978.

3. Pickett, A. E., Stress Corrosion Tests of Reactor Structural Materials in Simulated BWR Environments, NEDE-13179, Class 2, April, 1971.

TABLE 1

Composition of Type 304 Stainless Steel Test Material

C	Mn	P	S	Si	Ni	Cr
0.043	1.63	0.019	0.013	0.53	9.00	19.05

TABLE 2

Mechanical Properties of Type 304 Stainless Steel Test Material

Test Temperature	0.2% Y.S.	UTS	% R.A.	% El.
Ambient	235 MPa (34.1 ksi)	549 MPa (79.6 ksi)	81.8	74.8
288°C (550°F)	136 MPa (19.7 ksi)	426 MPa (61.8 ksi)	74.6	50.8

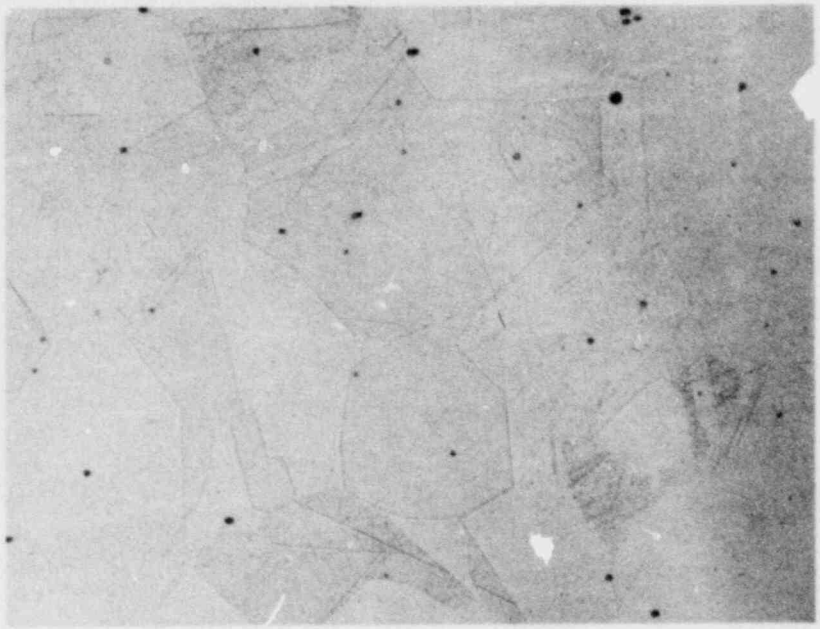
TABLE 3

DISTRIBUTION OF INTERGRANULAR PENETRATIONS AT FOUR LOCATIONS ACROSS THE WIDTH OF TWO BENT BEAM SPECIMENS

Section Polished	Number of Intergranular Penetrations[1]	
	Control	NS-1
Original	0	0
0.13 cm (0.05 in)	0	10
0.26 cm (0.10 in)	9	12
0.39 cm (0.15 in)	7	4

Notes:

(1) All penetrations were less than 1×10^{-3} cm (.0004 in).



250 X

FIGURE 1 - As-received microstructure of heat no. 78500

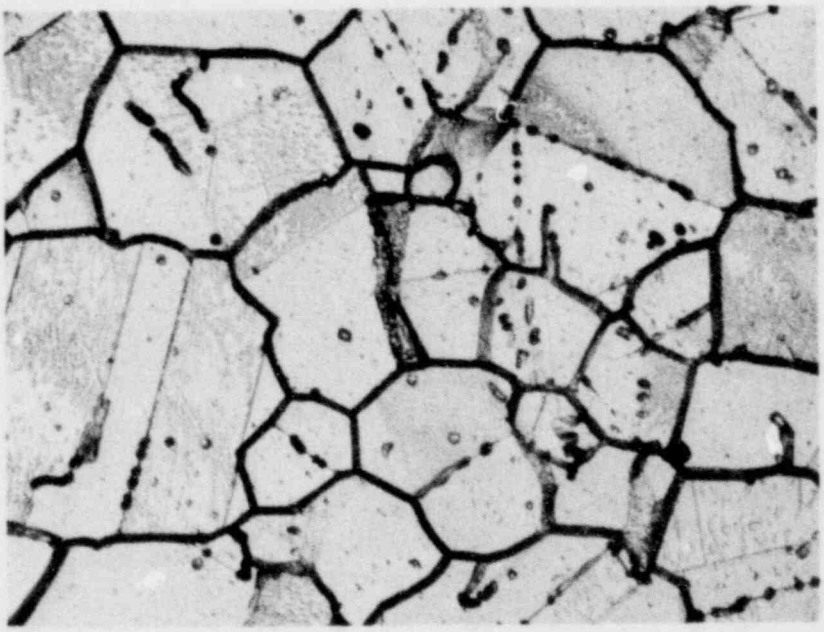


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FIGURE 2 - Response of heat no. 78500 to sensitizing for 50 hours at 675°C (1250°F); ASTM A 262, Practice A, etch.

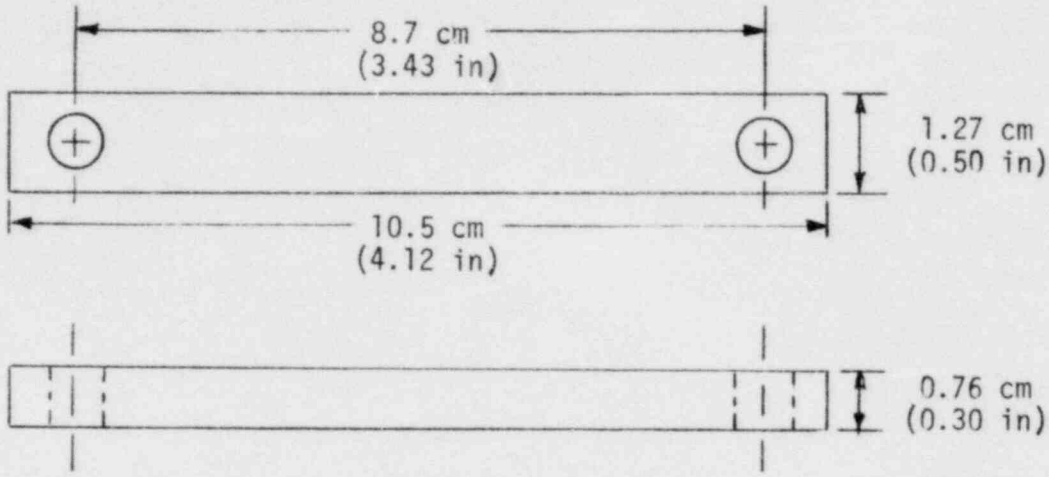


FIGURE 3 - Bent beam specimen drawing.

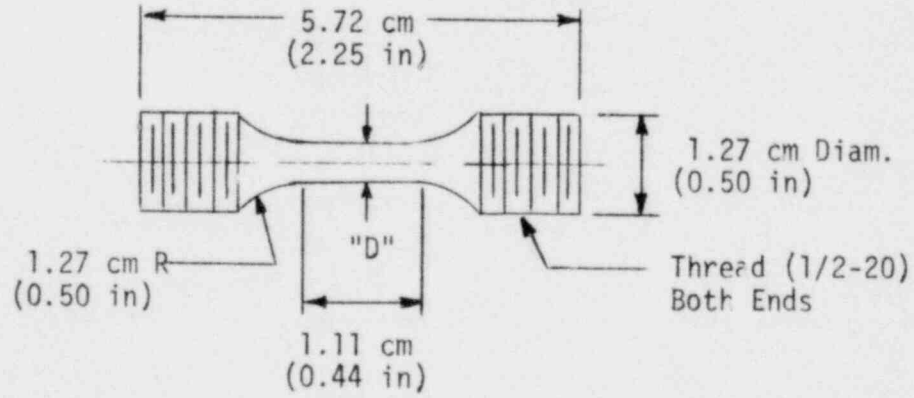


FIGURE 4 - Uniaxial tensile specimen drawing. Dimension "D" is adjusted to give desired stress level.

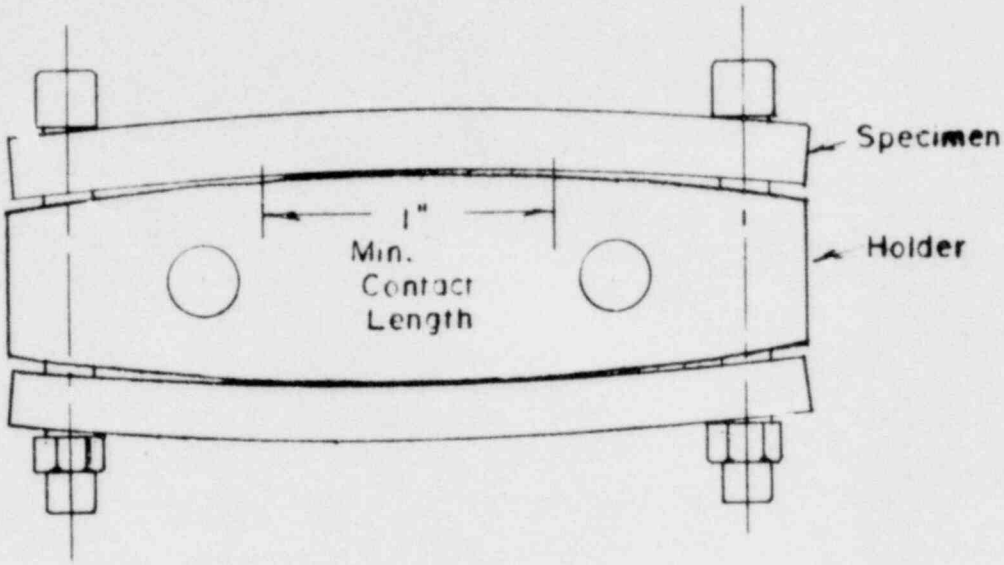


FIGURE 5 - Assembled bent beam specimen pair on stainless steel radius block.

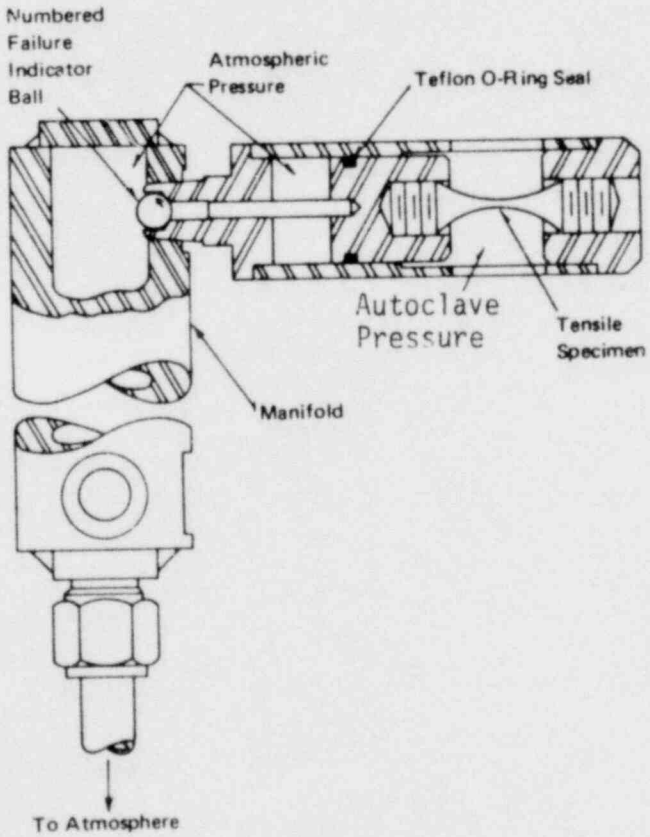
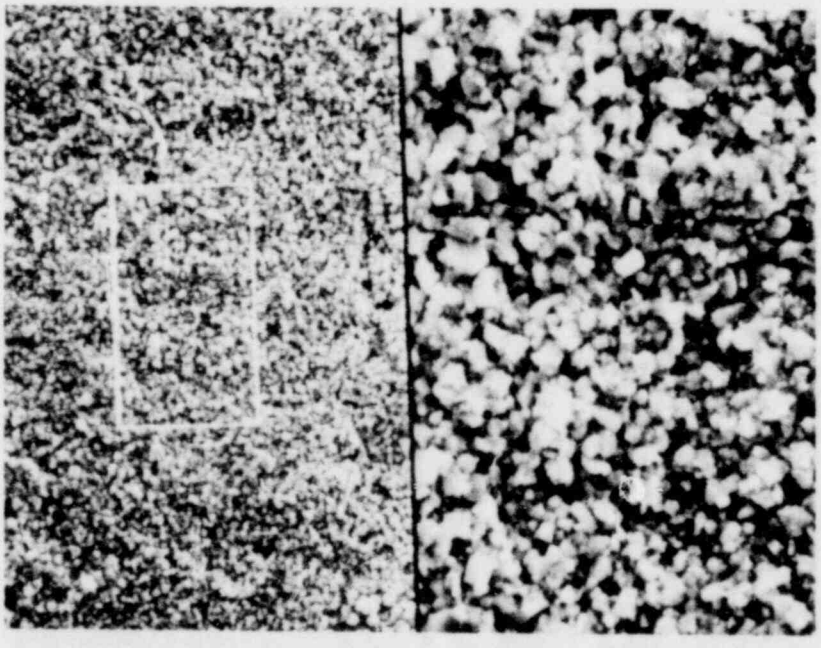


FIGURE 6 - Uniaxial tensile specimen loading module.

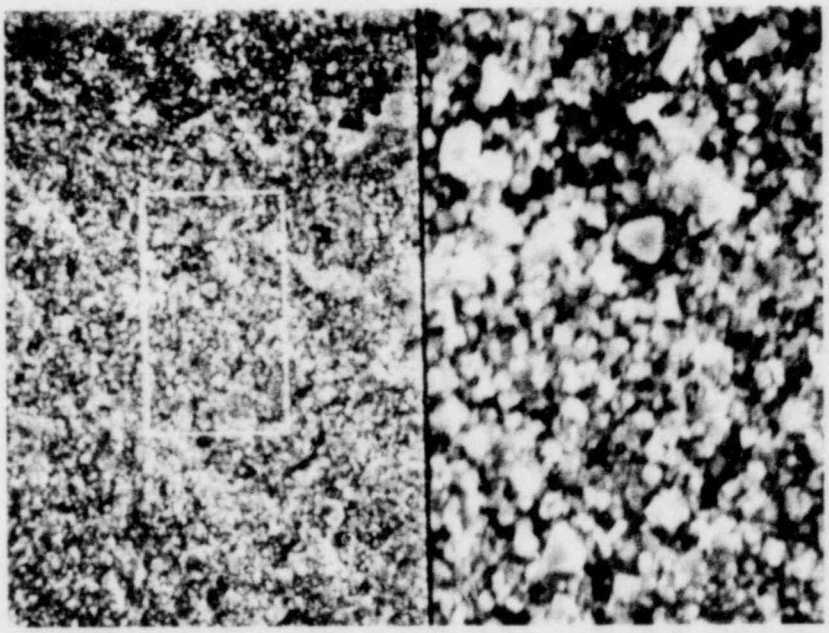
100 X



500 X 1500 X

FIGURE 7 - Scanning electron photomicrographs of surface of demineralized water control uniaxial tensile specimen exposed to BWR environment at 125% of 0.2% yield strength.

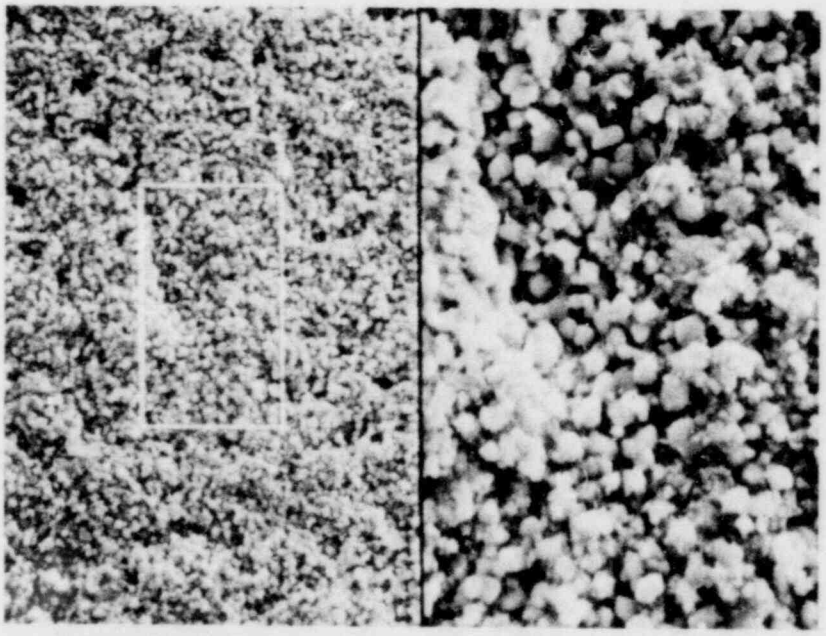
100 X



500 X 1500 X

FIGURE 8 - Scanning electron photomicrographs of surface of NS-1 uniaxial tensile specimen exposed to BWR environment at 125% of 0.2% yield strength.

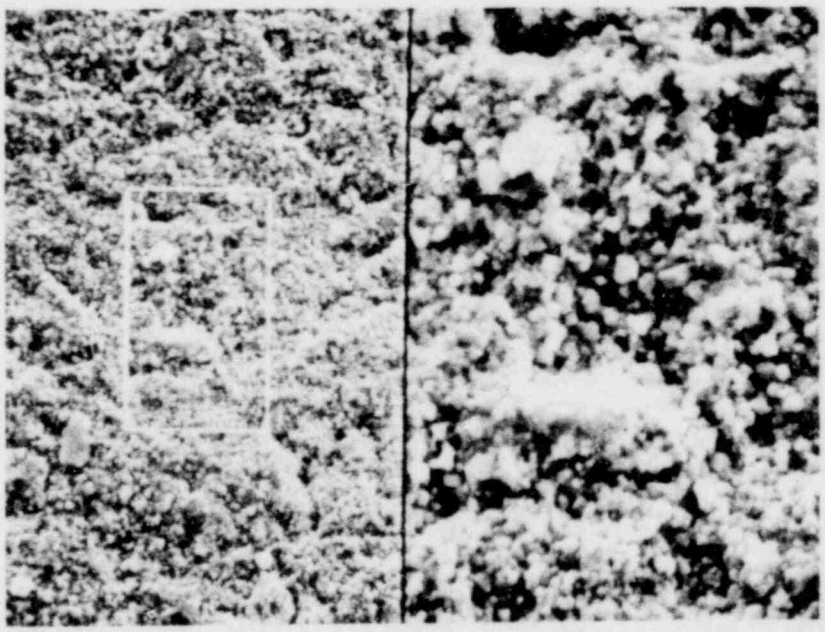
100 X



500 X 1500 X

FIGURE 9 - Scanning electron photomicrographs of surface of demineralized water control uniaxial tensile specimen exposed to BWR environment at 100% of 0.2% yield strength.

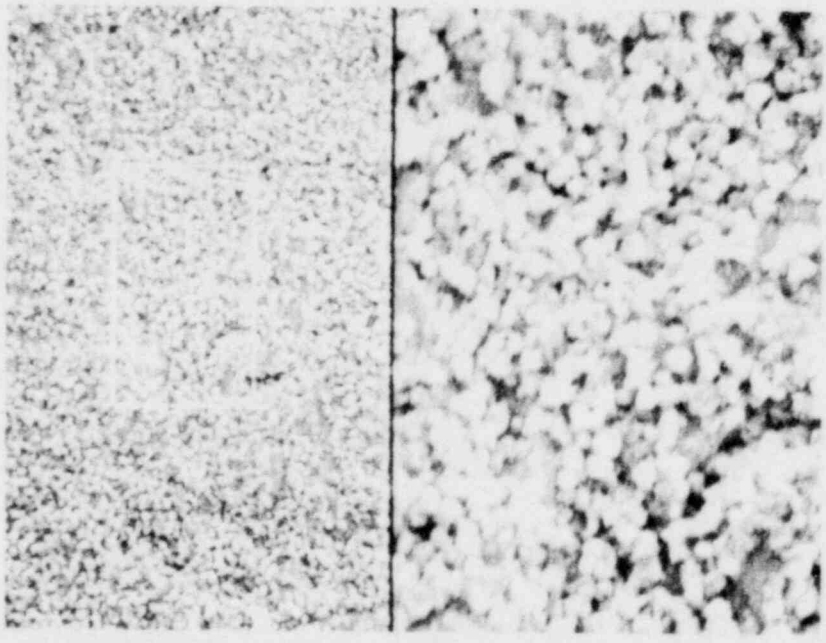
100 X



500 X 1500 X

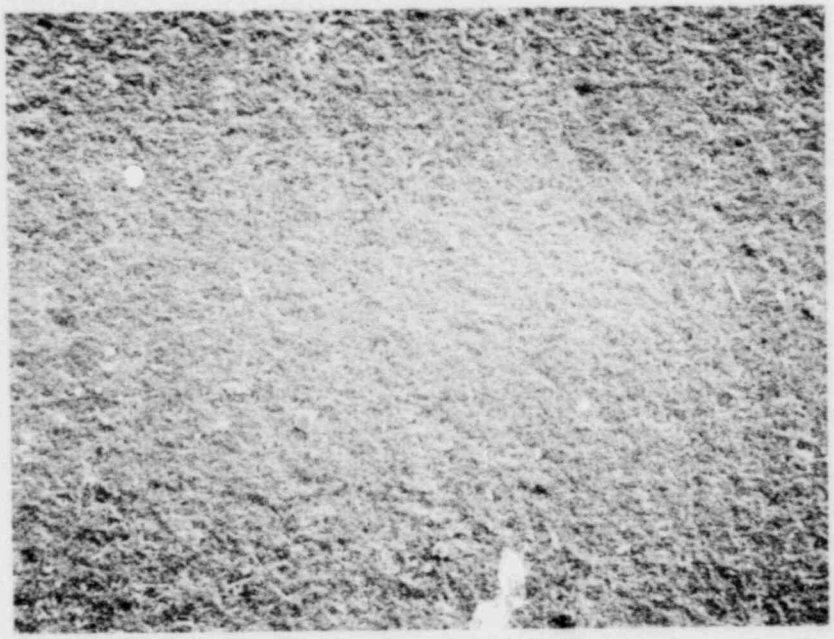
FIGURE 10 - Scanning electron photomicrographs of surface of NS-1 uniaxial tensile specimen exposed to BWR environment at 100% of 0.2% yield strength.

100 X

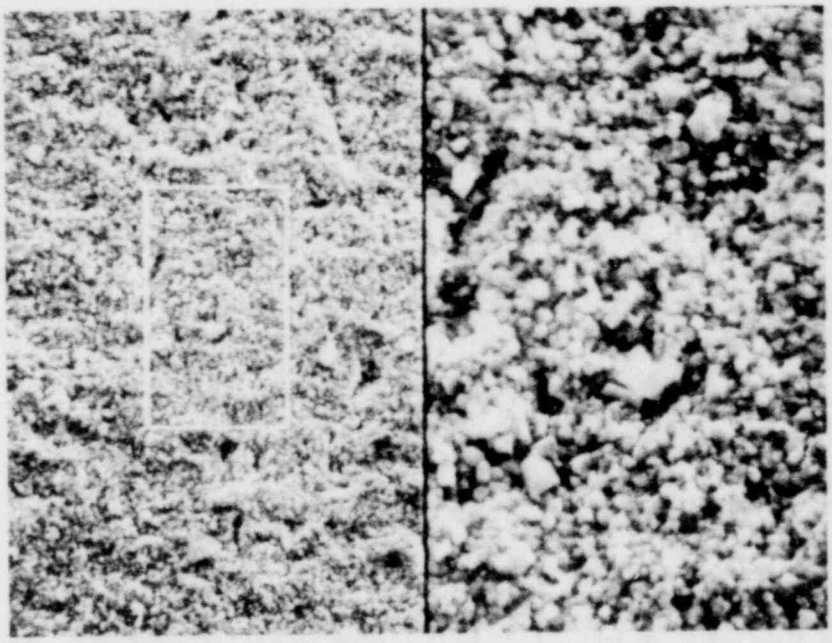


500 X 1500 X

FIGURE 11 - Scanning electron photomicrographs of surface of demineralized water control uniaxial tensile specimen exposed to BWR environment at 75% of 0.2% yield strength.



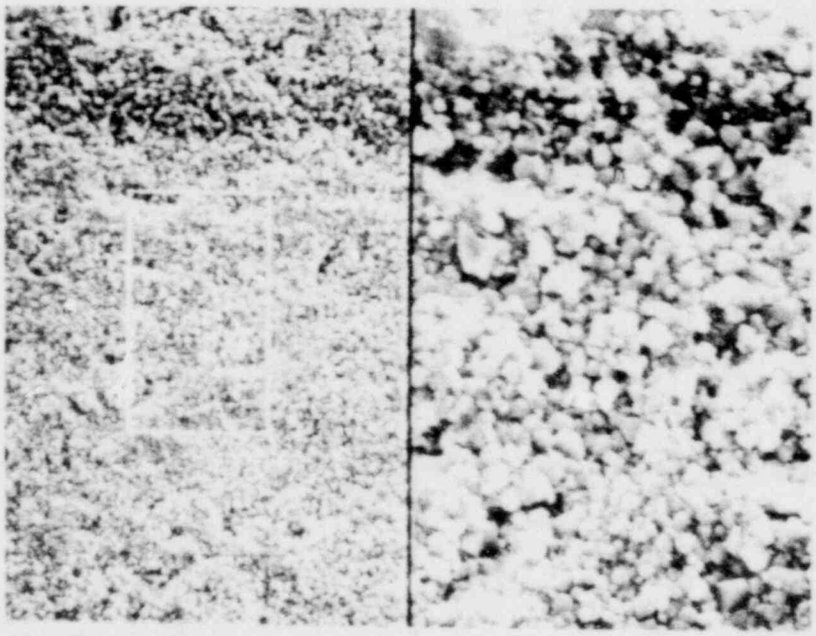
100 X



500 X 1500 X

FIGURE 12 - Scanning electron photomicrographs of surface of NS-1 uniaxial tensile specimen exposed to BWR environment at 75% of 0.2% yield strength.

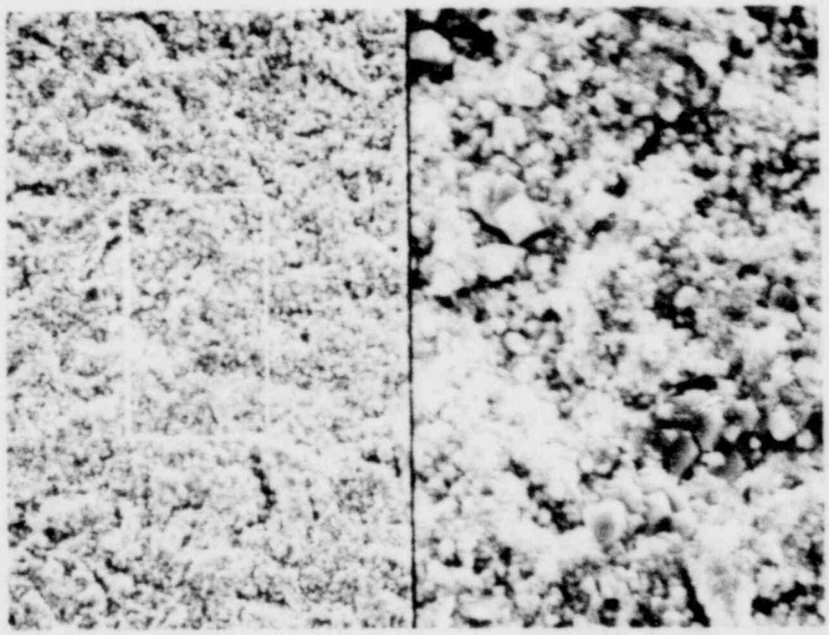
100 X



500 X 1500 X

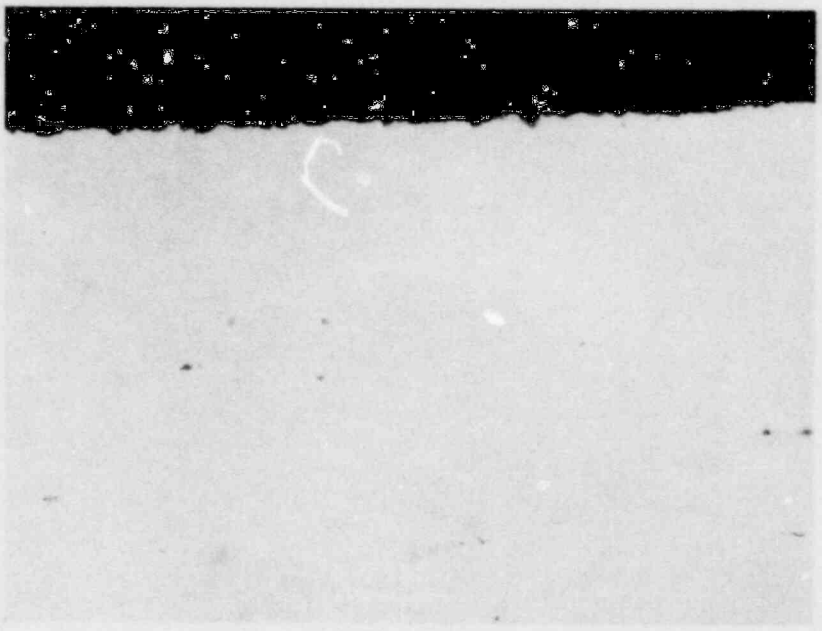
FIGURE 13 - Scanning electron photomicrographs of surface of demineralized water control bent beam specimen exposed to BWR environment at 2% strain.

100 X

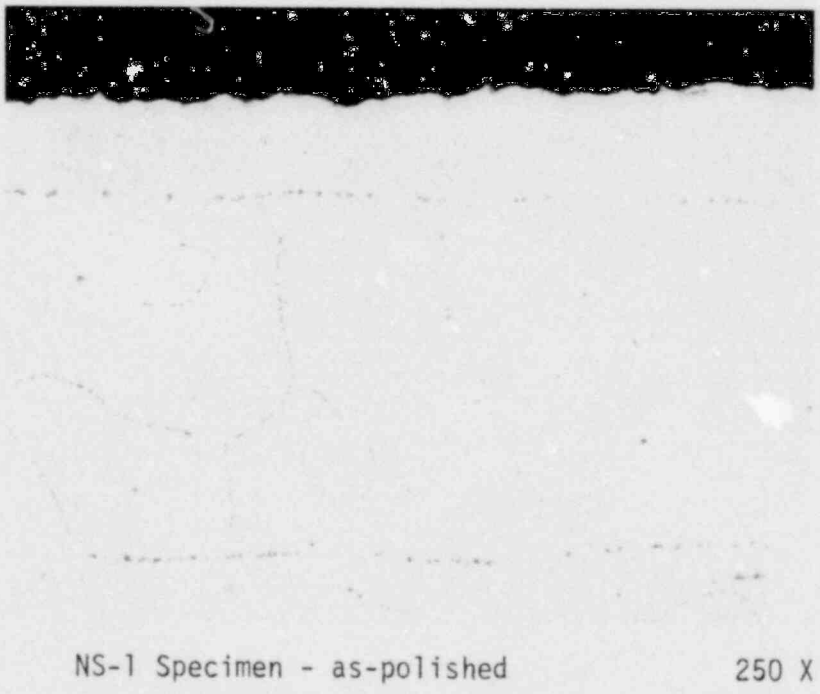


500 X 1500 X

FIGURE 14 - Scanning electron photomicrographs of surface of NS-1 bent beam specimen exposed to BWR environment at 2% strain.

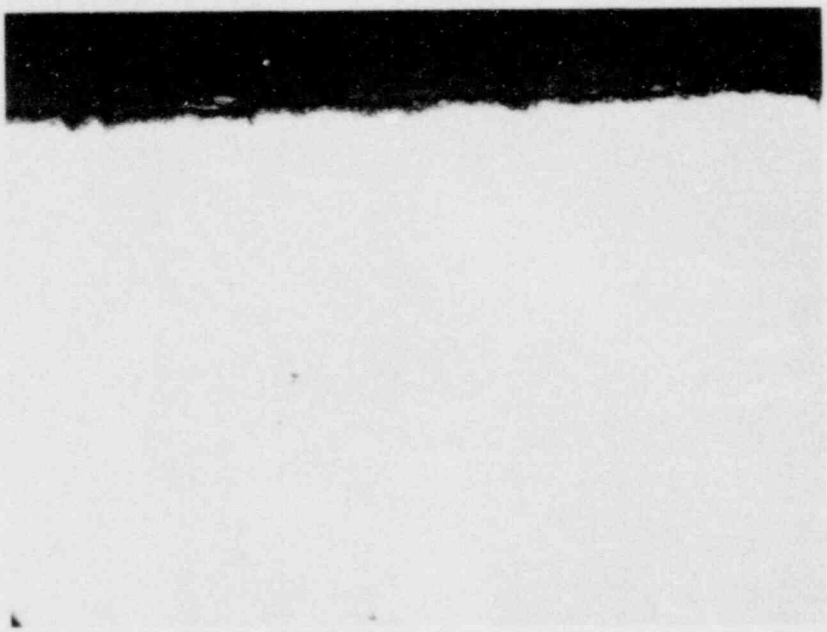


Control Specimen - as-polished 250 X

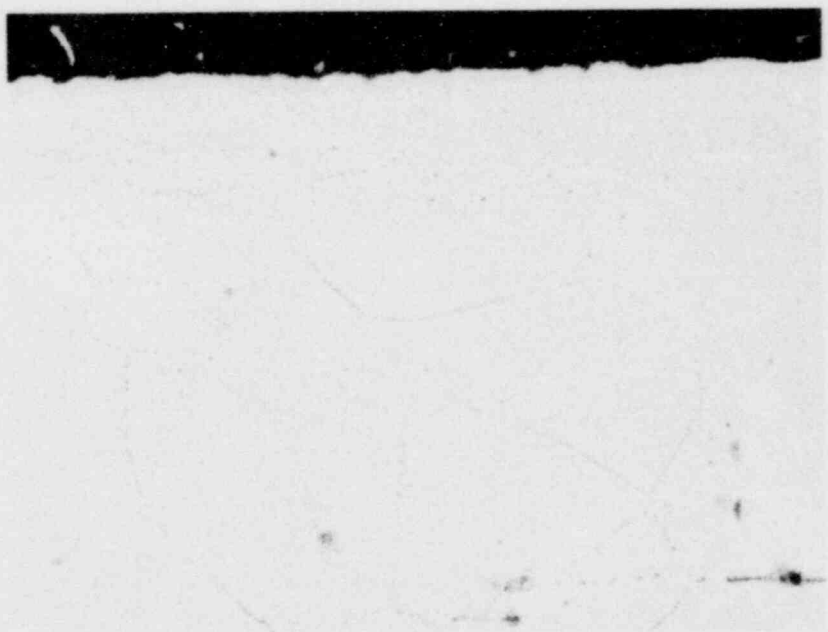


NS-1 Specimen - as-polished 250 X

FIGURE 15 - Longitudinal sections through demineralized water control and NS-1 uniaxial tensile specimens exposed to BWR environment at 125% of 0.2% yield strength.

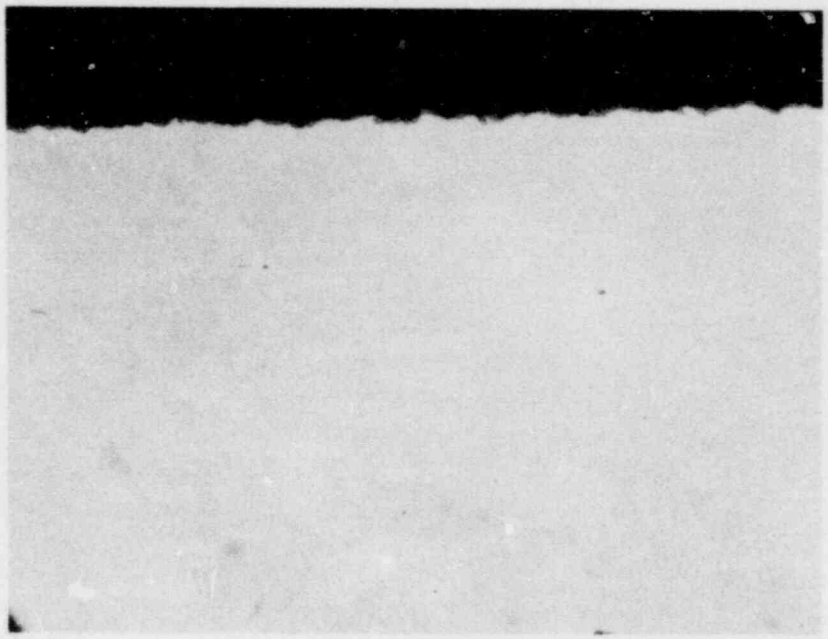


Control Specimen - as-polished 250 X



NS-1 Specimen - as-polished 250 X

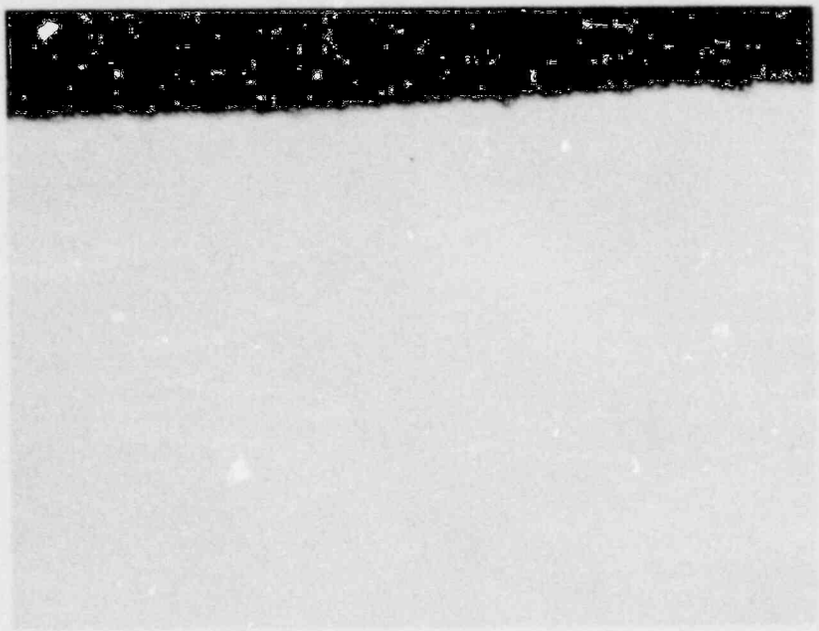
FIGURE 16 - Longitudinal sections through demineralized water control and NS-1 uniaxial tensile specimens exposed to BWR environment at 100% of 0.2% yield strength.



Control Specimen - as-polished 250 X

NS-1 Specimen - as-polished 250 X

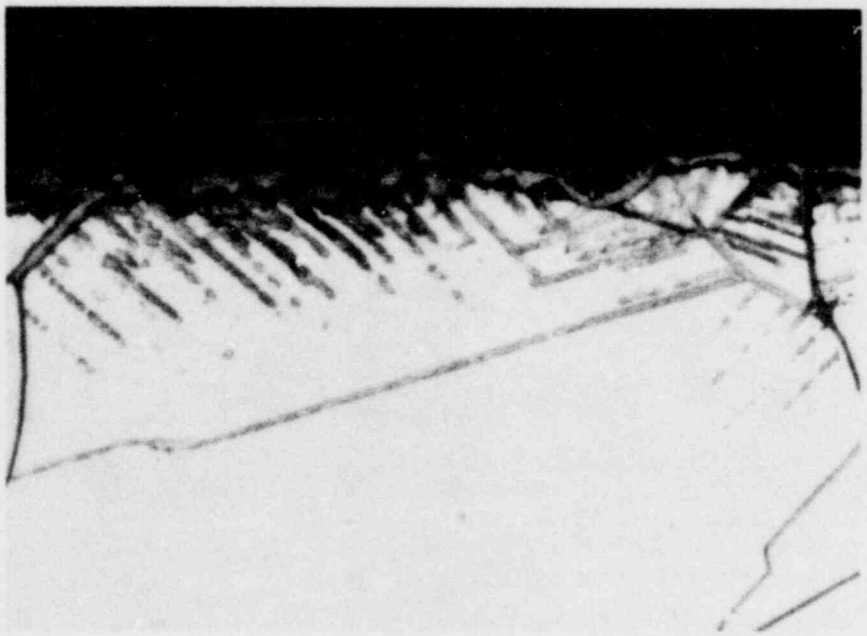
FIGURE 17 - Longitudinal sections through demineralized water control and NS-1 uniaxial tensile specimens exposed to BWR environment at 75% of 0.2% yield strength.



Control Specimen - as-polished 250 X

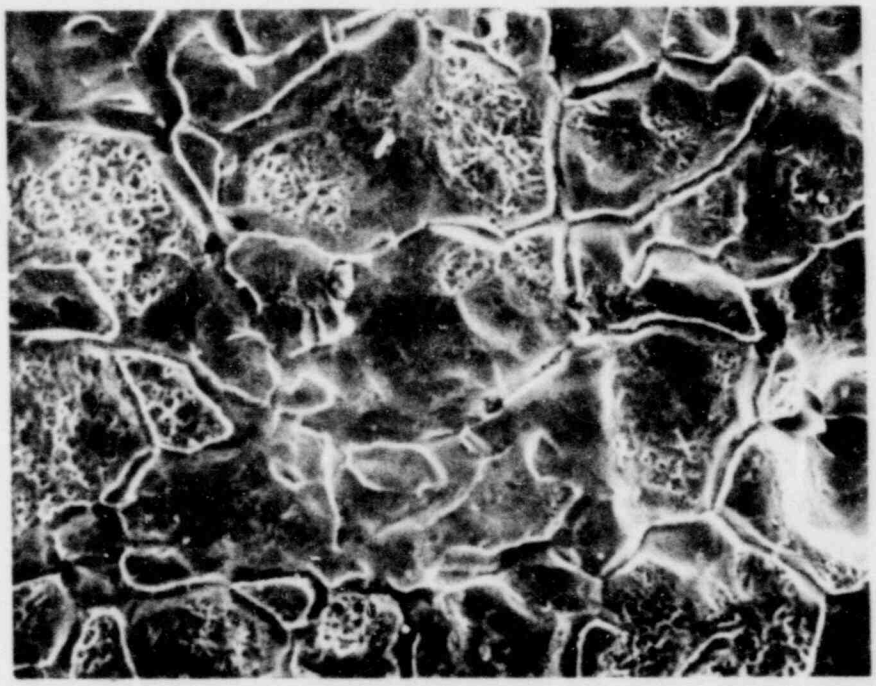
NS-1 Specimen - as-polished 250 X

FIGURE 18 - Longitudinal sections through demineralized water control and NS-1 bent beam specimens exposed to BWR environment at 2% strain.



800 X

FIGURE 19 - Lightly etched high magnification photomicrograph of tensile surface of NS-1 bent beam specimen. Note that intergranular penetrations are filled with oxides and that oxide film is continuous over the specimen surface.



300 X

FIGURE 20 - Scanning electron photomicrograph of inside diameter surface of pipe heat no. 78500 in as-received condition, showing intergranular attack.

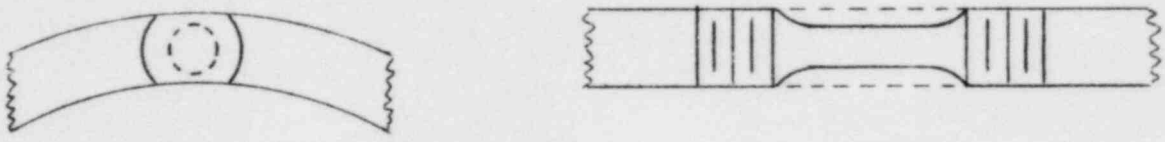
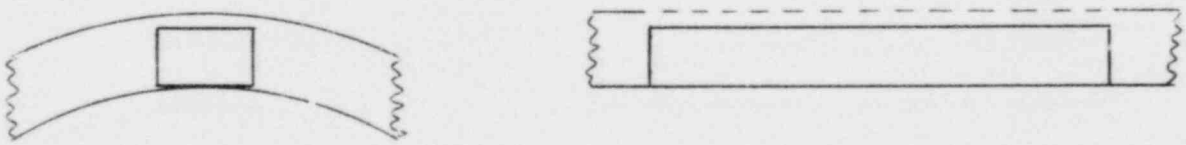


FIGURE 21 - Typical uniaxial tensile specimen location in pipe mid-wall.

22 - A

22 - B



22 - C

FIGURE 22 - Possible locations of bent beam specimens.
A - Ideal mid-wall; B - Outside diameter;
C - Inside diameter.