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INTERIM REPORT

NRC Research and Technical  
Assistance Report

## STRESS CORROSION CRACKING OF ALLOY 600 USING THE CONSTANT STRAIN RATE TEST\*

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INTRODUCTION

The most recent corrosion problems experienced in nuclear steam generators tubed with Inconel alloy 600 is a phenomenon labeled "denting." Denting has been found in various degrees of severity in many operating pressurized water reactors.<sup>(1)</sup> This denting or inward deformation of the steam generator tubing has been attributed to impurity-induced corrosion of the carbon steel in the crevice region of the tube-to-plate intersection.<sup>(2)</sup> Magnetite corrosion products which are trapped in the crevice occupy approximately twice the volume of the parent metal and create a force sufficient to deform the tubing. High strains and stresses imposed on the tubing as well as slow strain rates experienced during deformation provided conditions which have, in some cases, produced extensive intergranular stress corrosion cracking (SCC) originating

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from the primary side. These failures occurred at the deformation sites and near the top of the bundle in small radius U-bends which were ovalized by the inward deflection of the tube legs. It is not yet certain which specific factor was responsible for in-service cracking i.e., whether the slow straining caused SCC or if failures would continue in highly stressed areas if the active denting was arrested.

Laboratory investigations have shown that Inconel 600 exhibits intergranular SCC when subjected to high stresses and exposed to deoxygenated water at elevated temperatures.<sup>(3,4)</sup> However, the literature does not contain quantitative data, and does not show the effects of slow strain rates on the SCC of Inconel 600 in a deaerated aqueous media such as pure or primary water.

A research project was initiated at Brookhaven National Laboratory in an attempt to improve the qualitative and quantitative understanding of factors influencing SCC in high temperature service-related environments. An effort is also being made to develop an accelerated test method which could be used to predict the service life of tubes which have been deformed or are actively denting.

Several heats of commercial Inconel 600 tubing were procured for testing in deaerated pure and primary water at temperatures from 290°C to 365°C. U-bend type specimens were used to determine crack initiation times which may be expected for tubes where denting has occurred but is arrested and provide baseline data for judging the accelerating effects of the slow strain rate method. Constant extension rate tests were employed to determine the crack velocities experienced in the crack propagation stage and predict failure times of tubes which are actively denting.

#### EXPERIMENTAL

Twelve heats of Inconel 600 tubing were used in this investigation. Many of these heats had a microstructure thought to be susceptible to SCC based on prior experience and published literature.<sup>(5)</sup> Essentially, the grain boundaries

did not show much evidence of chromium carbide networks when electrolytically etched in phosphoric acid. Table 1 details the mechanical properties and chemical compositions of these materials. All of the compositions fall within the specifications for Alloy 600 and are typical of nuclear grade production with the exceptions of heats #6 and #12 which were purchased in the cold worked condition. It is also possible that the low carbon (0.01%) material would not be considered for current steam generator production. The recent 20 hour 700°C heat treatment that is now preferred by several manufacturers was not included in the tubing as purchased, although we did heat treat several specimens in this manner in the laboratory. The commercial tubing, therefore, is typical of material currently in service in operating plants. The various heat treatments that were used on the two lots of cold worked tubing in an effort to produce a susceptible structure are shown in Table 2.

Tests in pure deaerated water environments were conducted in one or two gallon 316 stainless steel pressure vessels under static conditions. Distilled deionized water, having a conductance of  $\leq 0.5 \mu\text{s}$  was deoxygenated by alternate pressure vent cycles with 3.4 MPa (500 psig) of high purity  $\text{N}_2$ . This was followed by evacuation of the vessel and then steaming off 10% of the water. The efficiency of this deoxygenating procedure was determined by using ASTM D888 Referee method A, which is a colorimetric indigo carmine analysis. The oxygen concentration was found to be in the range of 0 to 5 ppb.

Simulated primary water exposure of U-bend specimens were carried out in refreshed autoclave systems. The once through solution contained  $10^{-4}\text{M}$  LiOH and 10,000 ppm of B as boric acid and the  $\text{H}_2$  pressure in the solution reservoir was adjusted to 35 STP cc/Kg.

#### Constant Deflection Tests

Tube type U-bend specimens were prepared by splitting pieces of tubing on their longitudinal axis then bending with a modified tube bender. This produced a U-bend specimen with the original inside surface of the tubing in tension and

a 1.3cm (0.5") inside radius of bend. Standard type C-ring specimens were also prepared and stressed to either 95% or 110% of their yield strength. Heat treatments for the cold worked tubing and pickling were performed prior to bending the specimens and details of these procedures are listed in Table 2 and Table 3 respectively.

Duplicate U-bend and C-ring specimens were exposed to high purity deaerated water at test temperatures of 290°C, 325°C, 345°C and 365°C. Specimens were examined at two week intervals except for the 290°C test which was inspected every six weeks. Specimen exposures at 365°C and 345°C were terminated after 36 weeks and tests at the two lower temperatures and in simulated primary water environments are continuing.

#### Constant Extension Rate Test (CERT)

The CERT apparatus shown in Figure 1 is a modified version of one originally designed at the General Electric Company.<sup>(6)</sup> A linear variable differential transformer (L.V.D.T.) is used to monitor the specimen extension and a 907 Kg (2000 lb) load cell to measure the load. Both of these parameters are continuously recorded during the test. The extension rate range of this system is  $10^{-4}$  to  $10^{-9}$  cm sec.<sup>-1</sup>.

Tensile specimens were fabricated from tubing that was split along the longitudinal axis and then rolled very carefully to obtain flat pieces with a minimum reduction. Measurements showed that the rolling resulted in a one or two mil reduction from the original wall thickness, i.e. test pieces were about 3% cold worked during rolling. The specimens were machined in accordance with ASTM A370 and had .635 cm x 2.54 cm (.25" x 1") gauge section. The faces corresponding to the inside and outside wall of the tubing were not machined or polished. In order to examine the effect of the small amount of cold work, specimen designs have been selected for future tests where the flattening and rolling is eliminated.

## RESULTS

Duplicate U-bend specimens were tested in the as-received condition, and also with pickling or additional heat treatments. The additional heat treatments given to the cold worked materials are shown in Table 2. The heat treatments and pickling were applied before bending the specimens.

Five heats of material have failed by intergranular SCC in pure deaerated water at test temperatures of 365°C and 345°C. The failure times listed in Table 4 show that SCC is produced very rapidly at 365°C. Heat #2, which is regular production type steam generator tubing cracked as early as the first 2 week period without having received any additional heat treatments or pickling. The low carbon heats (#4 and #5) had longer times to failure at 365°C; however, this difference was not as pronounced at 345°C. It should also be noted that heat #5 is the only one to have cracked thus far in the test at 325°C which has now been operating for 36 weeks. Duplicate specimens have agreed reasonably well, but there are some exceptions, typical of the usual scatter that is often encountered in corrosion experiments of this nature.

Pickled specimens have shown a somewhat erratic correlation with their "as received" duplicates, and varied with the heat involved. Heat #2 appears to be indifferent to the effects of pickling whereas heat #10 cracked only in the pickled condition and the time to failure for heat #11 was markedly decreased by pickling. The low carbon materials (heats #4 and #5) cracked mostly when unpickled at 365°C, and mostly in the pickled condition at 345°C, also, the single specimen which cracked in the 325°C test was a pickled specimen from heat #5. The variations produced by pickling may or may not be related directly to the carbon level (#2=.05%, #4=.01%, #5=.01%, #10=.02%, and #11=.03%) and all its aspects will need to be further explored.

Heats #6 and #7 are susceptible to SCC in the heavily cold worked condition even at 290°C and stress levels of 95% y.s. and 110% y.s. C-rings prepared from these cold worked materials were the only ones which produced cracking even though all materials were exposed as C-rings in each of the tests. Heat treat-

ments of these cold worked materials produced a structure which did not fail by SCC after 36 weeks at 365°C.

Tests in primary water at 365°C have been initiated very recently and duplicate specimens from heat #2 have failed in the 3 to 5 week period. These results are very similar to those in pure water and in this respect agree well with CERT results discussed later in this paper.

Microscopic examination of the failed specimens showed that SCC was intergranular and generally along the axis of the U-bend near the apex. This would seem to indicate that a considerable hoop stress was applied during the bending process. Occasionally the cracks would deviate from this path and run for a short distance at nearly right angles before resuming the axial path. Micrographs of cracked specimens (Figs. 2, 3, and 4) and specimens which did not fail (Figs. 5 and 6) both show very little chromium carbide particles when etched electrolytically in phosphoric acid. Therefore, it appears that an accurate prediction of U-bend performance cannot be based on microstructural analysis alone.

The general temperature trends are shown in Figs. 7, 8 and 9. A  $T_F$  increase by a factor of up to 5 accompanies the 20°C drop from 365°C to 345°C. Tentatively, in the middle of the range covered by the sets of curves, the activation energy is 30 to 40 Kcal/mole; it will be better calculated when more points are obtained, so that we can ascertain the existence of a straight line semi-log plot and, if so, improve the value to be used for its slope. With the scatter and narrow temperature difference so far, it is not too surprising that the results cannot be more quantitative. Another aspect that will be clarified by more data points is the present trend for lower carbon samples to show a smaller temperature dependence, i.e. a lower activation energy.

#### Constant Extension Rate Tests (CERT)

Slow strain rate tests have proven to be a very accelerated test method. Materials which take many weeks to crack when tested as U-bends often fail in several days and frequently at lower strains in the slow strain rate tests.

Strain rates spanning a range of  $10^{-6} \text{ sec}^{-1}$  to  $10^{-8} \text{ sec}^{-1}$  have produced intergranular SCC in production material when exposed to either pure deaerated water or primary water environments. Table 5 lists the results of tests conducted thus far and compares these times to failure with U-bend type specimens.

Heat #4 was chosen to explore the temperature and strain rate effects because of the relatively long time needed to produce failure in this material when tested as U-bends. Figure 10 is the stress versus strain curves for this material when tested at  $365^{\circ}\text{C}$ ,  $345^{\circ}\text{C}$  and  $325^{\circ}\text{C}$  in pure deaerated water and  $\dot{\epsilon}$  about  $3 \times 10^{-7} \text{ sec}^{-1}$ . Failure by intergranular SCC was produced very rapidly at  $365^{\circ}\text{C}$  and the ductility increased as the temperature decreased. The fracture surfaces examined by SEM (Figs. 11, 12 and 13) showed intergranular SCC penetrating 1.15mm at  $365^{\circ}\text{C}$  0.80mm at  $345^{\circ}\text{C}$  and only 0.40mm at  $325^{\circ}\text{C}$ . Since the crack propagation rate diminished very rapidly with temperature, it was assumed that a longer exposure time, therefore a slower strain rate was necessary to produce significant intergranular penetration at the lower temperatures. The test at  $325^{\circ}\text{C}$  was repeated using a  $\dot{\epsilon} = 3.9 \times 10^{-8} \text{ sec}^{-1}$  and Figure 14 shows that the strain to failure was greatly decreased by using the lower strain rate. SEM examination of the fracture face (Fig. 15) shows approximately 2.5 times the intergranular penetration at the lower strain rate. It should be noted that the crack depths for these two specimens were proportional to the exposure times and the calculation of the crack velocities yielded very similar results. It has been shown for other materials that a portion of the crack velocity versus strain rate curves is fairly flat and therefore a crack velocity range exists that is relatively independent of strain rate.<sup>(7)</sup> It is apparent that both of the tests conducted at  $325^{\circ}\text{C}$  fall within this range.

The best approach to evaluate the severity of SCC is to calculate the crack velocities since this is independent of the material strength of the various heats and eliminates inconsistencies which affect methods such as reduction in area measurements where several cracks propagating on the same plane can introduce errors. An assumption was made that the cracking starts at about the



Figure 16 is a semi-log plot of crack velocity in millimeters per second against 1000 times the inverse of the absolute temperature. These are typical plots for obtaining activation energies for temperature dependent processes. It can be seen that the four data points for heat #4 give rise to a very good straight line. The least squares method shows a confidence factor of  $\gamma^2 = .99$  and an activation energy about 43 Kcal/mole.

Additional slow strain rate tests were carried out on heat #4 to determine the effect of environmental changes i.e., testing in a system constructed entirely of Inconel 600 as compared to the above mentioned tests which were performed in stainless steel systems and testing in primary water. Fig. 16 demonstrates that very little change occurred in the crack velocities due to either of these conditions. The data points fall well within the scatter band which may be expected for such tests. Crack velocity, for a specimen which was aged for 18 weeks in pure deaerated water at 365°C prior to testing in CERT is also shown in Fig. 15. This test was performed to evaluate structural changes or the presence of an oxide coating which may be present on a tube in service prior to denting. The data from the test agreed very well with the data for specimens that were not aged.

As previously mentioned, it was necessary to cold work the tubing to some extent when fabricating plate type tensile specimens from the production tubing. This caused some concern that the small amount of cold working may accelerate SCC. A single ligament specimen was fabricated from a section of heat #4 tubing without flattening or deformation. This specimen was tested in CERT at 365°C in pure deaerated water and Fig. 16 shows that the crack velocity was significantly lower than experienced by a flattened specimen tested under the same conditions. However, the secondary crack density for the tube type specimen was much greater than that of the plate type specimen and this could have effectively lowered the stress intensity on the primary fracture or the strain rate at a given point and produced a lower crack velocity. Future work will include dual ligament tube

type tensile specimens to further evaluate the effect of small amounts of cold working.

Several specimens from heat #2, which cracked very rapidly as U-bends in 365°C pure deaerated water, were tested in the slow strain rate test. Fig. 16 shows that these points fall on a line parallel to the data obtained for heat #4. This is a very good indication that a single mechanism controls the kinetics of the crack propagation stage and other heats of material are expected to show similar slopes.

Figure 17 shows a comparison of the stress-strain curves for heat #2 in the as received, mill annealed condition, and another that was subjected to a heat treatment of 20 hrs. in Ar at 700°C. This latter treatment is equivalent to the latest commercial method used to induce chromium carbide precipitation followed by chromium rediffusion into the denuded zones, and which is believed to provide resistance to SCC in deaerated high temperature water. The mill annealed material had approximately 50% intergranular failure, whereas the material after 700°C treatment showed a ductile fracture with only extremely shallow intergranular penetration. This is a very encouraging result, because the laboratory heat treatment was but a single step in what obviously was otherwise "adverse" processing in terms of SCC resistance. In production, we believe that the prior processing may be arranged to optimize the effects of the final 700°C heat treatment, and may well produce even greater resistance to this kind of SCC.

More tests are now planned with samples of commercial (700°C treated) tubing, and these will include a range of strain rates to obtain comprehensive data, including primary coolant conditions.

#### DISCUSSION

The ten heats of mill annealed Inconel 600 tubing used in this program are typical of nuclear grade production, however, only five of these heats have shown evidence of intergranular stress corrosion cracking when U-bend specimens

were exposed to pure deaerated water at high temperatures. It is difficult to establish what differences exist between these heats that account for the fact that some are susceptible while others appear to be immune. Earlier studies<sup>(5)</sup> have shown that a susceptible structure is generally associated with carbide free grain boundaries and more recent work<sup>(8)</sup> has demonstrated that semi continuous grain boundary precipitates are very beneficial in preventing SCC in caustic environments. Electrolytic etching in phosphoric acid showed that all of the materials used in this program were relatively free of carbide precipitates in the grain boundary regions. The susceptibility of this alloy, therefore, cannot be judged on micro structural analysis alone. Small variations in processing history which occur within a mill or different mills must play an important role.

U-bend specimens were fabricated from tubing that varied both in outside diameter and wall thickness. The outside diameters ranged from 22mm (.875") to 14mm (.562") and the wall thickness from 2.4mm (.095") to 1.1mm (0.45"). Forming tubes with these different dimensions into reverse type U-bends, all having the same inside radius of bend, produced axial stresses and hoop stresses which varied considerably from heat-to-heat. The walls of the larger diameter thin wall tubing flared more when bending than that of the heavier walled materials thus providing a larger hoop stress. It is the hoop stress that appears to be dominant in the SCC of these U-bends since the cracks run along the axis of the tubing. The magnitude of these stresses are not known at this time and photoelastic techniques are being used to obtain this information. The results from the U-bend tests can, therefore, not be examined quantitatively until the stress levels are known.

Additional data are expected from continuing tests in pure water environments at 325°C and 290°C. These data will aid in determining the existence of a straight line semi-log plot of failure time versus the inverse absolute temperature. The failure time for U-bend specimens from heat #5, when tested at 365°C, 345°C and 325°C, indicate that a straight line relationship may exist. If this is established to include operating steam generator temperatures, a con-

siderable amount of laboratory exposure time can be eliminated by testing at the highest temperatures and extrapolating to operating conditions.

While the effect of pickling does appear to be somewhat erratic at the highest test temperature, most of the susceptible heats show that this treatment does have an adverse result.

The advantages of using the slow strain rate test to produce SCC has been well documented. This method eliminates the long exposure times necessary to initiate cracking in constant deflection type tests and ultimately results in specimen fracture either by SCC or some other mechanism. This test also more appropriately duplicates the conditions experienced by steam generator tubes that are undergoing active denting.

A strain rate range of  $10^{-6}$   $\text{sec}^{-1}$  to  $10^{-8}$   $\text{sec}^{-1}$  has been effective in producing intergranular SCC. As the test temperature was lowered, it was necessary to decrease the strain rate to provide a substantial amount of intergranular SCC. The test of one low carbon heat of material in pure water at  $325^{\circ}\text{C}$  show that cracking could be initiated at strain rates of  $2.9 \times 10^{-7}$   $\text{sec}^{-1}$  and  $3.9 \times 10^{-8}$   $\text{sec}^{-1}$ , however, the lower strain rate provided the longer exposure time necessary to produce deep cracking. This test clearly showed that the crack depth was dependent on the exposure time during straining and that although the crack depths were significantly different the calculation of the crack velocities gave nearly identical results.

All of the fractured surfaces were examined by SEM to determine the presence of intergranular SCC even though most specimens exhibited varying degrees of secondary cracking. Intergranular penetrations originated in the specimen faces corresponding to the original ID or OD of the tubing rather than the machined surfaces. They were not, however, confined to a particular side; the inside surface of the tubing appears to be as susceptible as the outside.

The small amount of cold working (3%) needed to fabricate plate type tensile specimens from tubing seems to be beneficial in accelerating SCC in susceptible materials. The one specimen that was tested without flattening or

rolling had a significantly lower crack velocity than a plate type specimen tested under the same conditions. However, materials which did not fail in U-bend tests also showed no sign of SCC after cold working and exposing to the constant extension rate. It appears at this time that although cold working does decrease the failure time of susceptible materials, it does not in itself promote SCC. Additional tests are planned to investigate the effects of cold working.

It is necessary to define the stress or strain level at which crack initiation occurs before crack velocities can be calculated. Since tests are scheduled to determine this value, but not completed, the yield point of the material was chosen as the crack initiation time. Crack velocities were calculated by dividing the maximum intergranular crack depth by the straining time from yield to failure. These results showed very good agreement between specimens from one heat when tested in either pure or primary water. These data provided a straight line relationship of the log crack velocity versus the inverse absolute temperature. The data from two heats of material (heat #2 & #4) that were tested at various temperatures produced parallel results thus indicating identical activation energy. If this straight line relationship exists from 365°C to operating temperatures for the crack initiation stage, as indicated by the U-bend tests, and also for the crack propagation stage, rapid testing at elevated temperatures will be a valid approach to evaluating new heats of material scheduled for service. Testing of materials currently in service will also provide some insight into the life expectancy of operating units.

#### CONCLUSIONS

1. Intergranular SCC of mill annealed Alloy 600 in pure, deaerated water can be produced readily. Failures occurred in less than 2 weeks in some highly plastically deformed U-bends exposed at 365°C.
2. Failure times in primary water environments appear to be unchanged from those in pure water.

3. Constant extension rate tests produce intergranular SCC in Alloy 600 after significantly shorter exposure times and frequently at lower strains than U-bend specimens.
4. Strain rates of  $10^{-6}$  sec<sup>-1</sup> produce intergranular SCC in pure water at 365°C but rates of  $10^{-8}$  sec<sup>-1</sup> or lower are needed to provide sufficient time for appreciable crack depth in a susceptible material at 290°C.
5. Cold working decreases the crack initiation time and accelerated the crack velocity of susceptible heats of Alloy 600.
6. There is a distinct variation in the degree of resistance to SCC from one heat to another.
7. A heat treatment at about 700°C can increase the resistance of an otherwise susceptible alloy very substantially.
8. Data appear to be sufficiently quantitative to hold promise for setting up equations that can predict SCC lifetimes under given conditions.

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Table 1 Mechanical Properties and Chemical Composition of Materials.

MATERIAL IDENTIFICATION	ULTIMATE STRENGTH psi	YIELD POINT psi	ELONGATION %	ROCKWELL HARDNESS	CHEMICAL ANALYSIS										
					C	Mn	Al	S	Si	Ni	Cr	Ti	Cu	Fe	Co
Ingot Heat #1					.08	.44		.003	.17	74.1	15.6		.36	9.27	
Tubing Heat #2	104,000	56,000	39.	R <sub>B</sub> 86	.05	.28	.19	.003	.24	73.94	15.69	.20	.33	9.47	.05
Tubing Heat #3	100,000	48,000	43	R <sub>B</sub> 83.5	.03	.30	.13	.007	.15	74.82	15.48	.16	.29	8.55	.06
Tubing Heat #4	91,285	46,580	48	R <sub>B</sub> 79-82	.01	.26		.004	.10	76.74	15.10		.21	7.56	
Tubing Heat #5	94,085	54,930	44	R <sub>B</sub> 83.5	.01	.28		.006	.08	75.59	15.76		.31	7.94	
Tubing Heat #6 (unannealed)					.029	.28		.003	.20	75.40	15.78		.01	7.99	.045
Plate Heat #8	96,000	37,750	45	R <sub>B</sub> 78-81	.05	.25		.004	.07	75.73	14.57		.29	9.04	
Tubing Heat #9	100,000	48,000	44.5	R <sub>B</sub> 81	.03	.28	.23	.006	.11	75.87	15.14	.25	.23	8.33	.05
Tubing Heat #10	99,000	48,000	45.5	R <sub>B</sub> 79	.02	.30	.32	.0006	.14	75.95	15.17	.26	.22	8.19	.84
Tubing Heat #11	97,000	51,000	42.5	R <sub>B</sub> 80	.03	.30	.17	.004	.18	74.64	15.18	.17	.34	9.33	.04
Tubing Heat #12* (cold worked)	122,990	119,060	10	R <sub>C</sub> 24											
Tubing Heat #13	96,300	44,200	47	R <sub>B</sub> 84	.03	.19		.001	.29	75.48	15.93		.02	7.52	.051

\* Heat #5 with 40% cold work

Table 2 Treatment of Cold Worked Tubing before Forming into U-bend Specimens

Specimen from Heat #6 were given the following treatments then exposed to 365°C deaerated H<sub>2</sub>O for 24 weeks without any evidence of I.G.S.C.C.

15 min in H<sub>2</sub> @ 1710°F  
15 min in H<sub>2</sub> @ 1710°F + pickle  
15 min in H<sub>2</sub> @ 1775°F  
15 min in H<sub>2</sub> @ 175°F + pickle

Specimens from Heat #12 were given the following treatment then exposed to 365°C deaerated H<sub>2</sub>O for 22 weeks without any evidence of I.G.S.C.C.

10 min in H<sub>2</sub> @ 1600°F  
" " " " " " " + pickle  
10 min in H<sub>2</sub> @ 1625°F  
" " " " " " " + pickle  
10 min in H<sub>2</sub> @ 1650°F  
" " " " " " " + pickle  
15 min in H<sub>2</sub> @ 1710°F  
" " " " " " " + pickle  
15 min in H<sub>2</sub> @ 1740°F  
" " " " " " " + pickle  
15 min in H<sub>2</sub> @ 1775°F  
" " " " " " " + pickle  
15 min in H<sub>2</sub> @ 1800°F  
" " " " " " " + pickle



Table 3 HNO<sub>3</sub>-HF Pickling Solution

Constituents	Requirements
HNO <sub>3</sub> (sp. gr. = 1.33)	275 parts by volume
HF (40%)	50 parts by volume
H <sub>2</sub> O	65 parts by volume
Temperature	122°F to 140°F
Time	15 min.

Table 4 U-bend Test Results

Heat #	Specimen Type	Test Temperature (°C)	Additional Treatments	<u>Specimens Failed</u> Specimens Tested	Average Time to Failure (Weeks)	
2	Tube U-bend " " "	365	As Received	3/4	2	
			Pickled	2/2	2	
			15 min in H <sub>2</sub> @1775°C	1/1	7	
			15 min in H <sub>2</sub> @1775°F + Pickle	1/1	1	
		345	As Received	2/2	14	
			Pickled	2/2	16	
4	Tube U-bend	365	As Received	2/2	11	
		345	Pickled	2/2	10	
5	Tube U-bend	365	As Received	4/4	12	
			Pickled	1/4	9	
		345	As Received	2/2	16	
			Pickled	2/2	13	
6	Tube U-bend C-Rings	325	As Received	1/2	29	
		365	As Received	2/2	1	
		365	As Received	4/4	1	
		345	As Received	4/4	1	
10	Tube U-bend	290	As Received	4/4	<9	
		365	Pickled	2/2	2	
11	Tube U-bend	365	345	Pickled	2/2	5
			As Received	1/2	13	
12	Tube U-bend C-Ring	365	Pickled	2/2	4	
			As Received	3/3	1	
			365	As Received	4/4	1
		345	As Received	4/4	5	

Table 5 Constant Extension Rate Test Results

Heat No.	Test Temperature °C	Plate Type Tensile Specimens Exposed to Pure Deaerated H <sub>2</sub> O in Stn. Stl. Vessels. Exceptions Noted Below	Strain Rate sec <sup>-1</sup>	% Strain to Failure	Intergranular Crack Depth (mm)	Crack Velocity mm sec <sup>-1</sup>	Time to Failure (Weeks)	
							Cert	U-bends
2	365		2.8x10 <sup>-7</sup>	13.8	0.58	1.18x10 <sup>-6</sup>	0.8	2
2	365	Specimen heat treated 20 hrs in Ar @700°C	3.0x10 <sup>-7</sup>	35.5	0.08	6.78x10 <sup>-8</sup>	2.0	>26
2	325		5.2x10 <sup>-8</sup>	32.1	0.44	8.18x10 <sup>-8</sup>	10.2	>36
4	365		2.6x10 <sup>-7</sup>	14.5	1.15	2.60x10 <sup>-6</sup>	0.9	11
4	365	Unflattened tube type specimen	3.4x10 <sup>-7</sup>	33.0	0.73	7.82x10 <sup>-7</sup>	1.6	
4	345		3.0x10 <sup>-7</sup>	21.8	0.80	1.35x10 <sup>-6</sup>	1.2	10
4	345	Simulated primary water	3.0x10 <sup>-7</sup>	27.7	1.14	1.38x10 <sup>-6</sup>	1.5	
4	345	Test conducted in Inconel 600 vessel	2.7x10 <sup>-7</sup>	28.0	1.29	1.58x10 <sup>-6</sup>	1.7	
4	325		2.9x10 <sup>-7</sup>	42.0	0.40	3.31x10 <sup>-7</sup>	2.4	>36
4	325		3.9x10 <sup>-8</sup>	12.0	0.95	4.44x10 <sup>-7</sup>	5.1	>36
4	325	Specimen aged 18 wks. in D.I. H <sub>2</sub> O @365°C	4.95x10 <sup>-8</sup>	16.9	1.33	4.98x10 <sup>-7</sup>	5.6	
4	290		5.0x10 <sup>-8</sup>	30.0	0.15	2.87x10 <sup>-8</sup>	9.9	>63
5	365		6.36x10 <sup>-8</sup>	Not pulled To Failure 41	0.81	1.29x10 <sup>-6</sup>		
5	365		2.0x10 <sup>-6</sup>		0.09	4.41x10 <sup>-7</sup>	0.3	12

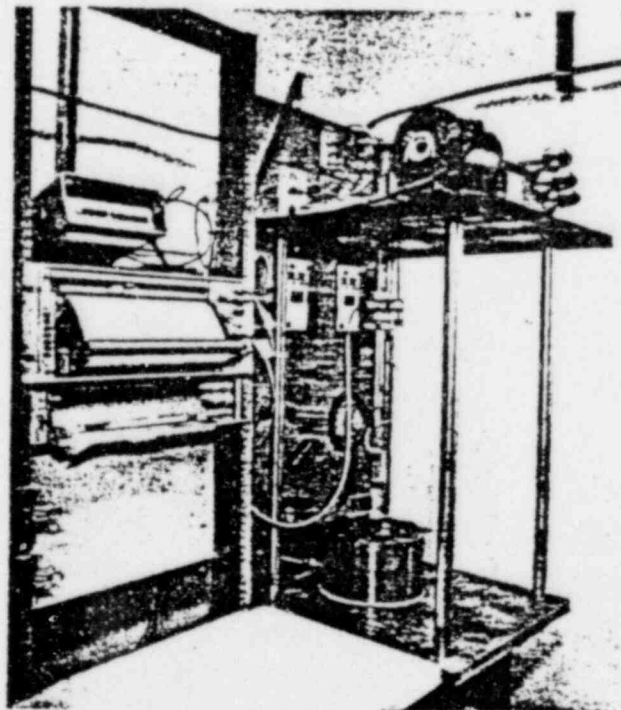
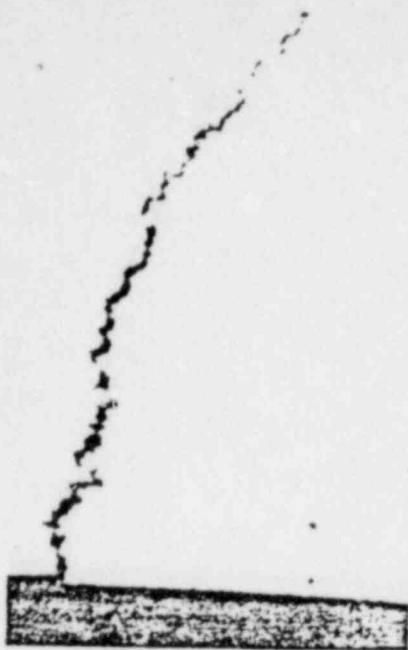


Figure 1 Constant Extension Rate Test



UNETCHED

68X



5% Nital

ELECTROLYTIC ETCH

350X



8:1 Phosphoric acid/H<sub>2</sub>O

ELECTROLYTIC ETCH

350X

Figure 2 Micrographs of U-bend Specimen from Heat #2 after 2 Weeks Exposure @3365°C



UNETCHED

68X



5% Nital

Electrolytic Etch

350X



8:1 Phosphoric acid/H<sub>2</sub>O

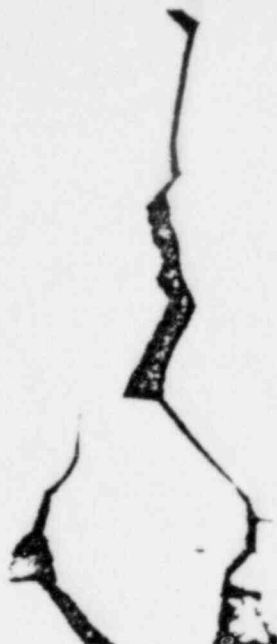
Electrolytic Etch

Figure 3 Micrographs of U-bend Specimen from Heat #4 after 5 Weeks Exposure @3345°C



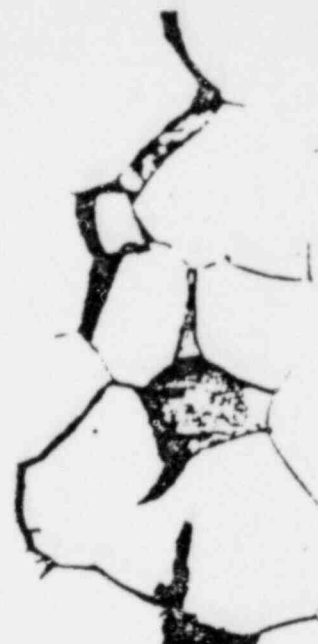
UNETCHED

68X



5% Nital  
ELECTROLYTIC ETCH

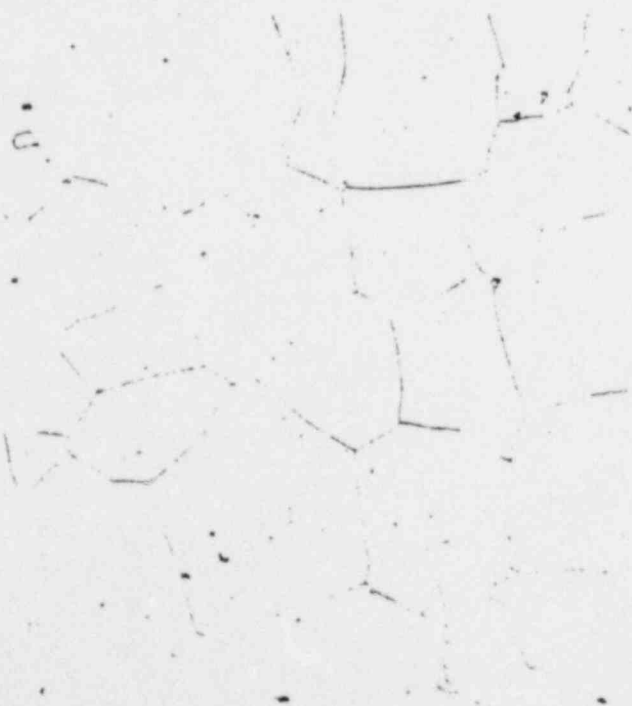
350X



8:1 Phosphoric acid/H<sub>2</sub>O  
ELECTROLYTIC ETCH

350X

Figure 4 Micrographs of U-bend Specimen from Heat #5 after 10 Weeks Exposure @365°C



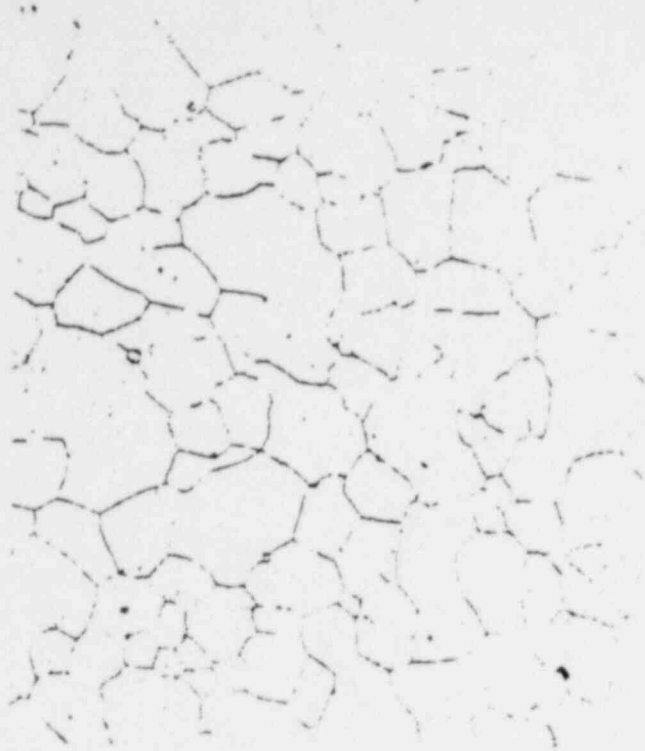
5% Nital  
ELECTROLYTIC ETCH

350X

8:1 Phosphoric acid/H<sub>2</sub>O  
ELECTROLYTIC ETCH

3 X

Figure 5 Structure of Material from Heat #3 Uncracked after 36 Weeks Exposure at 365°C



5% Nital  
Electrolytic Etch  
350X

8:1 Phosphoric acid/H<sub>2</sub>O  
Electrolytic Etch  
350X

Figure 6 Structure of Material from Heat #13 Uncracked after 30 Weeks Exposure @ 365°C

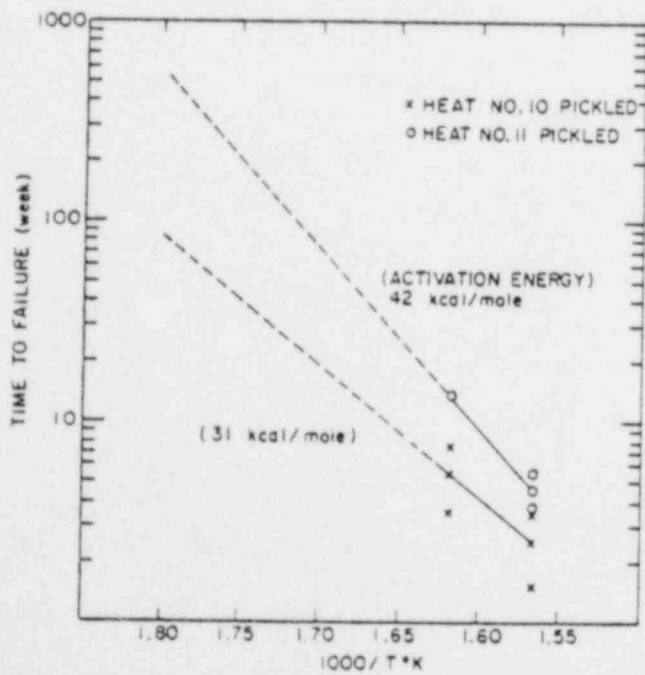


Figure 7 Failure Time vs. Temperature for SCC of U-bend Specimens in Pure Deaerated Water

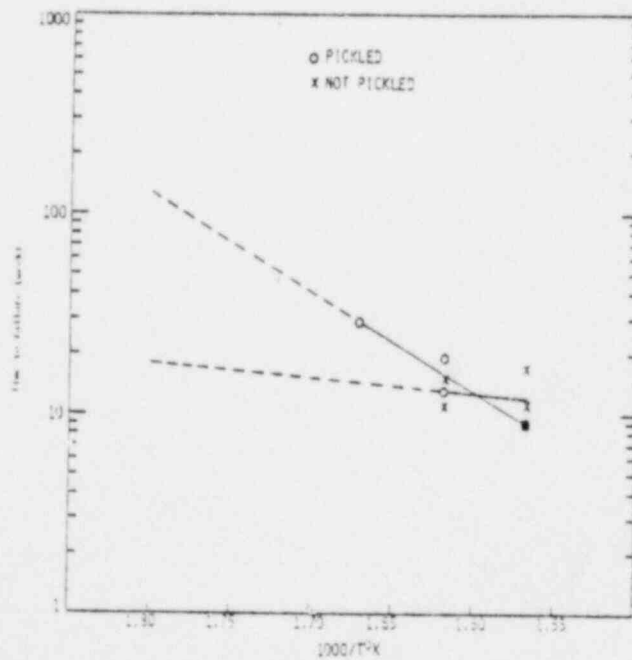


Figure 8 Failure Time vs. Temperature for SCC of Low Carbon (.01%) Heat #5 U-bend Specimens in Pure Deaerated Water

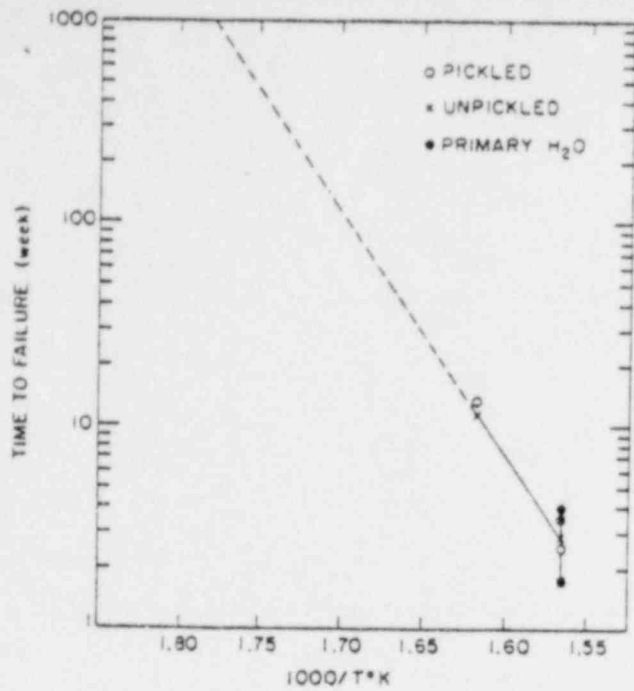


Figure 9 Failure Time vs. Temperature for SCC of Heat #2 U-bend Specimens in Pure and Primary Water



10X Photo. of Fracture Surface

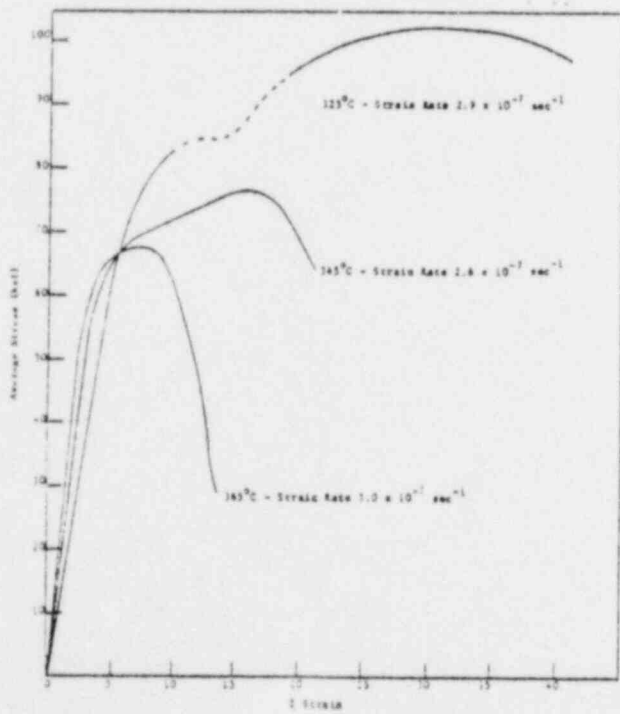
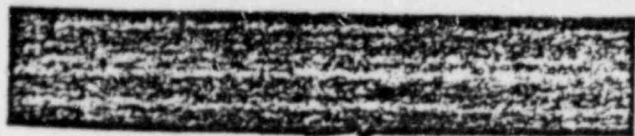


Figure 10 Stress-Strain Curve for Heat #4 in D.I. H<sub>2</sub>O

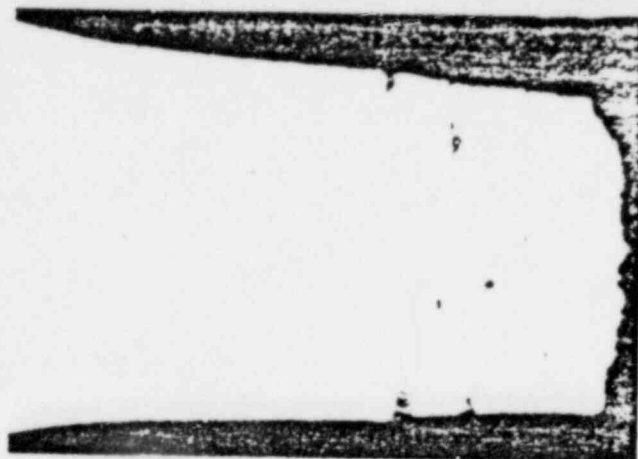
1 X S.E.M. of Fracture Surface



Figure 11 Heat #4 Fractured in Constant Extension Rate Test 3.35°C with a Strain Rate of  $3.0 \times 10^{-2} \text{ sec}^{-1}$



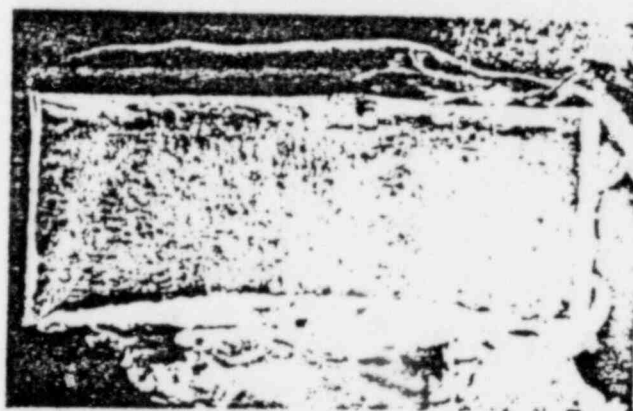
3X Photo of Fractured Gauge Section



8.5X Photo of Fractured Gauge Section



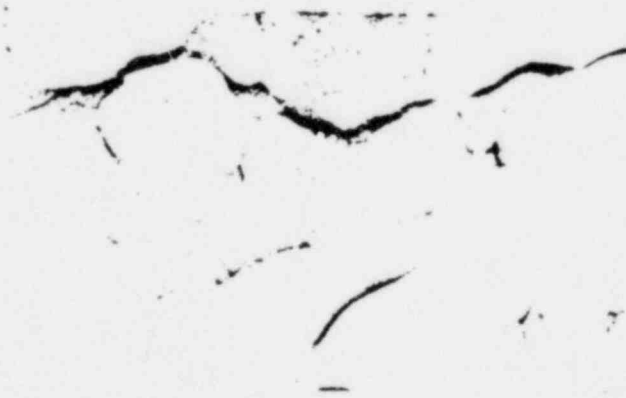
15X S.E.M. of Fracture Face



15X S.E.M. of Fracture Face



500X S.E.M. of Intergranular Area



500X S.E.M. of Intergranular Area

Figure 12 Heat #4 Fractured in Constant Extension Rate Test 8345°C with a Strain Rate of  $2.6 \times 10^{-7} \text{ sec}^{-1}$

Figure 13 Heat #4 Fractured in Constant Extension Rate Test 8325°C with a Strain Rate of  $2.9 \times 10^{-7} \text{ sec}^{-1}$

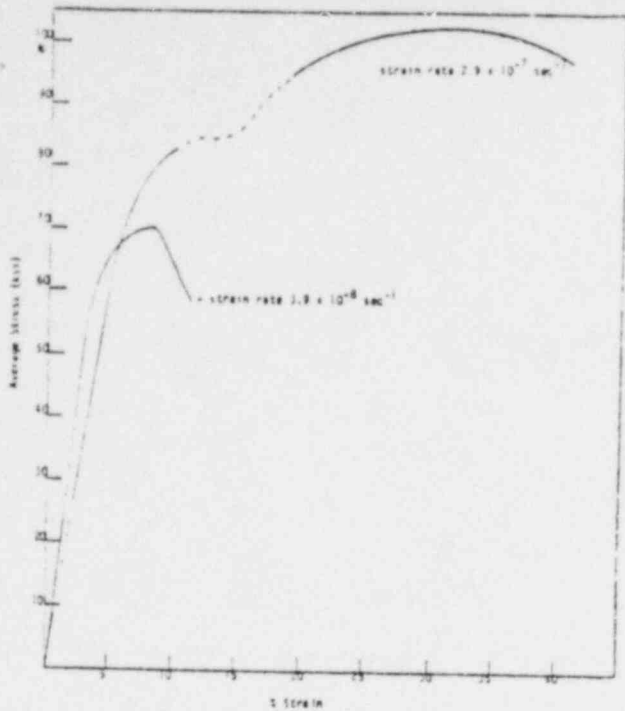


Figure 14 Stress-Strain Curve for Heat #4 in D.I. H<sub>2</sub>O @325°C

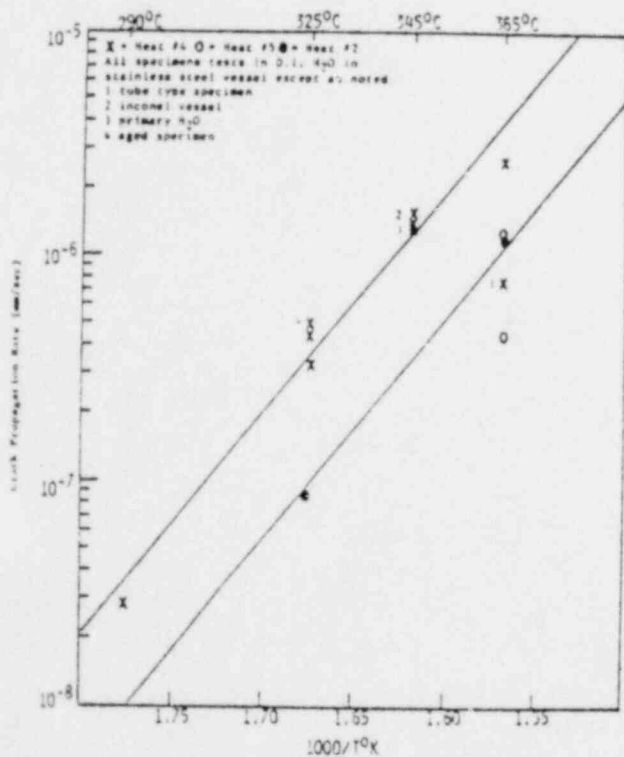
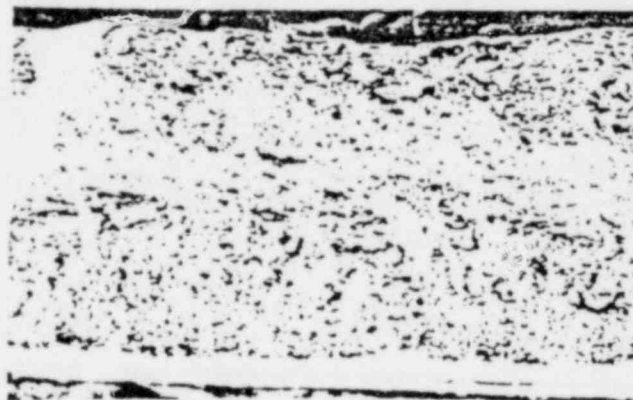


Figure 16 Effect of Temperature on Crack Velocity for Materials Tested in C.E.R.T.



7.5X Photo of Fractured Gauge Section



20X S.E.M. of Fracture Face



500X S.E.M. of Intergranular Area

Figure 15 Heat #4 Fractured in Constant Extension Rate Test @325°C with a Strain Rate of  $3.9 \times 10^{-8} \text{ sec}^{-1}$

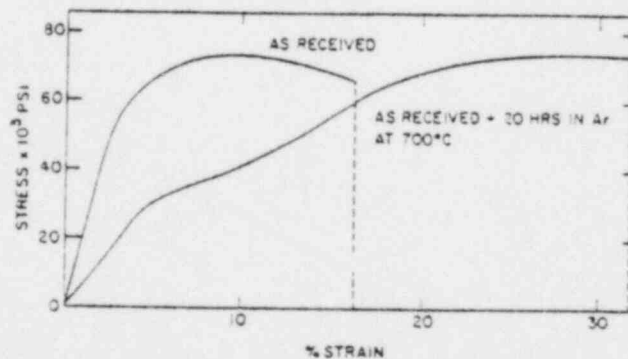


Figure 17 Effect of 700°C 20 hour Heat Treatment on Heat #2 Strained  $3.3 \times 10^{-7} \text{ sec}^{-1}$  in Pure Water at 365°C