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PRELIMINARY

NRC Research and/or Technical Assistance Report

Accession No. ________

Contract Program or Project Title: Environmentally Assisted Cracking in LWR Systems

Subject of this Document: Same as above.

Type of Document: Monthly Progress Report

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U.S. Nuclear Regulatory Commission
Washington, D. C. 20555

PRELIMINARY

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March 8, 1982

Dr. J. Muscara
Office of Nuclear Regulatory Research
Materials Engineering Branch
U.S. Nuclear Regulatory Commission
Mail Stop 1130SS
Washington, D.C. 20555

Dear Joe:

A copy of the January monthly report for our program on "Environmentally Assisted Cracking in LWR Systems" is enclosed. If you have any questions regarding this report, please contact either myself or any of the Principal Investigators.

Sincerely yours,

Bill

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Materials Science Division

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Monthly Progress Report

Contractor: Argonne National Laboratory

Program: Environmentally Assisted Cracking in LWR Systems

189a: A2212

B&R No: 60-19-01-30 (NRC)
40-10-01-06-1 (DOE)
8M443 and 8M454 (ANL)

Reporting Period:
1-1-83 thru 1-31-83

Task Manager:
W. J. Shack

Financial Summary:

	Baseline	Actual	Monthly Variance	Cumulative Variance
October	$183K	$183K	$ -	$ -
November	192K	192K	-	-
December	219K	200K*	19K	19K
January	204K	174K	30K	49K
February	170K			
March	170K			
April	204K			
May	164K			
June	164K			
July	204K			
August	164K			
September	202K			

*This number was reported incorrectly last month.

Subtask A: Leak Detection and NDE (D. S. Kupperman)

Work on modifying the acoustic leak detection facility is continuing. The frame to support the new pipe section (containing four laboratory grown cracks) has been installed. The cracks are now being welded into the new pipe section. The second field IGSCC does not show much leakage under low applied stresses (<2000 psi). Additional tests will be carried out at higher stress levels.

Two waveguide systems for field installation are being tested. Modifications are being made to provide the most effective waveguide system. One system is a "quick connect" type which uses spring loading to press the rounded waveguide tip to the pipe surface. The waveguide is attached to a plate which is strapped on the outside of the reflective insulation. In the other system the waveguide tip passes through the insulation and is screwed into a plate strapped on the pipe outer surface.

Six cracks in two 6-in. pipe sections recovered from an operating BWR have been located by dye penetrant techniques. The samples have been sectioned and decontaminated to a level that allows work to be carried out in a conventional laboratory environment. The cracks found are skewed (2), longitudinal (2), and circumferential (2).

A welded cast stainless steel pipe (28-in. O.D.) has been received from PNL. We have demonstrated, in a blind test, that longitudinal velocity of sound measurements can be used to differentiate between isotropic and anisotropic (long columnar grains) microstructures. In this case normal incidence longitudinal waves at 1 MHz were employed. Results were confirmed by PNL.

Subtask B: Analysis of Sensitization (J. Y. Park)

EPR values are being measured on Types 304 and 316 NG SS specimens which were heat treated at 500-750°C for 10-130 min in order to determine seeding treatments for LTS studies. The seeding treatment at 700°C for 10 min produces too much scatter (because the thermal mass of the sepcimens significantly affects the thermal history) or too large EPR values (>2 C/cm^2) as reported previously. An increase in EPR value was observed for a Type 304 SS (Heat No. 30956) specimen with a 500°C/24 h treatment after a seeding treatment at 650°C/10 min in the mill-annealed condition, while EPR value did not increase for a specimen with a prior solution heat treatment (1050°C/0.5 h) before the seeding treatment. This is in agreement with the general observation that low temperature sensitization kinetics are faster in the mill-annealed condition than those in the solution-annealed condition.

Studies on the effect of DOS on IGSCC susceptibility are continuing. IGSCC propagation rate measurements have been performed for LTS-treated Type 304 SS (Heat No. 10285) 1TCT specimen in water with 8 ppm oxygen at 289°C and 8.3 MPa. The specimen was furnace sensitized at 700°C/10 min followed by an additional 500°C/24 h heat treatment. The EPR value of a companion coupon was 4 C/cm^2. Crack propagation rates under a variety of loading conditions were reported previously. During the present reporting period, tests have been carried out at $R = 0.7$, $f = 2 \times 10^{-3}$ Hz and $K_{max} = 30$ $MPa \cdot m^{1/2}$. Preliminary analysis shows a propagation rate of 4×10^{-10} m/s. The construction of two additional CERT facilities was completed. Calibration of extension rate as a function of motor speed was carried out. The extension rates in the gage section of CERT specimens varied linearly with respect to motor speeds and were relatively independent of load in the range of steady load beyond yield. In addition to the simple tension CERT tests, cyclic tests can be run under load control. The specimens are electrically isolated from the rest of the system to permit better control of electrochemical experiments and use of the electric potential drop technique for crack length determination. Fabrication of 4-in. diameter Type 304 SS (Heat No. 53319) pipe weldments with the remedy treatments (IHSI, HSW, LPHSW and CRC) was completed. Specimens were out from the weldment for LTS studies. ASTM A262-A tests are being performed in the as-welded condition before low temperature aging.

Subtask C: Crack Growth Rate Studies (J. Y. Park and W. J. Shack)

Crack growth rate tests have been continued for a Type 304 SS (Heat No. 10285 EPR = 15 C/cm^2) 1TCT specimen in water with 8 ppm oxygen at 289°C and 8.3 MPa (1200 psi). Growth rates at maximum stress intensities (K_{max}) ranging from 30 to 34 $MPa \cdot m^{1/2}$, load ratios (R) of 0.5, 0.94, and 1, and frequencies (f) of 0, 2×10^{-3}, and 1×10^{-1} Hz have been reported previously. The cyclic loading tests have been performed using sawtooth waveforms with a loading-to-unloading time ratio of 99. The current series of tests will establish (at a fixed K) the effect of R ratio and frequency on the growth rate, and whether these effects can be understood in terms of the crack tip strain rate. During the present reporting period, the tests have been carried out at K_{max} = 28-29 $MPa \cdot m^{1/2}$, R = 0.6 and f = 2×10^{-3} Hz. The crack has propagated 0.7 mm during a 341-h period at an average rate of 5.6×10^{-10} m/s. The tests are being performed at R = 0.7 and f = 2×10^{-3} Hz. Crack propagation rate measurements for Type 304 SS (Heat No. 10285) to date are summarized in Table 1.

The investigation of the electric potential drop method for crack length measurement has been continued. A.C. potential drop measurements have been made for a growing crack in a Type 304 SS 1TCT specimen under a fatigue load in air at room temperature. A sensitivity of 1.2 volts/amp/in was obtained at crack lengths (a/w) between 0.25 and 0.32. Temperature dependence was less than 2.6×10^{-3} in/°C and long-term stability better than 1×10^{-3} in/day was observed. The calibration is now being extended to a wider range of crack lengths.

<u>Subtask D:</u> <u>Non-environmental Corrective Actions</u> (P. S. Maiya and W. J. Shack)

CERT experiments on Type 316 NG (Heat No. P91576) in chloride-containing water environments (8 ppm O_2) have established that the material becomes susceptible to TGSCC at a strain rate of 2×10^{-6} s^{-1} and 289°C after heat treatments at 650°C/2 h or 700°C/4 h. Whether the prior furnace heat treatment of the specimens is essential for the occurrence of TGSCC (in a manner similar to that observed for IGSCC in conventional Types 304 and 316 SS) remains to be established. TGSCC adversely affects CERT properties such as failure time and reduction in area just like IGSCC. The dependence of the TGSCC crack growth rates on strain rate in this environment is similar to that for IGSCC growth, and the results to date can be described by a power law i.e. $\dot{a} = 1.17 \times 10^{-7} (\dot{\varepsilon})^{0.29}$ m/s. A comparison of the transgranular crack growth rates (in 316 NG) with intergranular crack growth rates (observed in sensitized Type 316 SS) in this environment showed clearly that the transgranular crack growth rate is approximately 1/10th of the intergranular crack growth rate over the range of strain rates used (2×10^{-6} to 1×10^{-7} s^{-1}). These experiments are being continued to investigate whether SCC susceptibility can occur under impurity environments which are consistent with the operating limits in BWRs as specified in Regulatory Guide 1.56 (e.g., 0.2 ppm O_2 + 0.2 ppm Cl^-).

Subtask E: Evaluation of Environmental Corrective Actions (W. E. Ruther, W. K. Soppet, and T. F. Kassner)

The crack growth experiment on Type 304 stainless steel CT specimens (Heat No. 30956) in high-purity water with 8 ppm oxygen (289°C) continues. During January the test was conducted at a K_{max} of 48 $MPa \cdot m^{1/2}$, R = 0.95, and a frequency of 8×10^{-3} Hz. The crack in the more heavily sensitized specimen (EPR = 20 C/cm^2) grew at a linear rate of 15 mm/y over an 800-h period. No crack growth occurred in the EPR = 2 C/cm^2 and EPR = 0 specimens during this period. K_{max} has been increased to 60 $MPa \cdot m^{1/2}$ for the specimen sensitized to an EPR value of 20 C/cm^2.

The second crack growth experiment with the same heat of Type 304 SS in water containing 0.1 ppm $SO_4^=$ (as H_2SO_4) and 0.2 ppm oxygen (289°C) also continues. The loading conditions are K_{max} = 28 $MPa \cdot m^{1/2}$, R = 0.95 and f = 8×10^{-2} Hz. Even with fatigue precracks, no crack growth has occurred in 625 h. Measurements of the oxygen levels in the outflow of the autoclave indicate that because of oxygen scavenging the actual oxygen levels are less than 0.2 ppm. The gas mixture used to aerate the water is being altered to correct this.

Results from CERT experiments performed in January are given in Table II. These tests provide additional information on the effect of dissolved oxygen, hydrogen, and sulfate impurities on the SCC susceptibility of sensitized Type 304 SS. In the A-series of experiments, we are evaluating the relative effect of pH and sulfate concentration (at a dissolved oxygen concentration of 0.2 ppm) on the SCC behavior of this heat of material.

The performance of a 1.85-liter refreshed titanium autoclave system for high-temperature pH and corrosion-potential measurements in simulated BWR-quality water has been evaluated at 285°C and 8.3 MPa pressure. The titanium system has several advantages relative to a stainless steel autoclave that was being used for these experiments (viz., better control of the dissolved oxygen concentration, less interference from corrosion-product species, and more rapid replacement of the solution due to a smaller volume in the autoclave).

The problem of emf drift in the 0.1 M KCl/Ag/AgCl external reference electrodes, which are being used to monitor the corrosion and redox potentials, has been identified. The seal produced by the thin-wall heat-shrinkable teflon (TFE) tubing, between the porous zirconia frit and the electrolyte compartment of the reference electrode, degrades during thermal and pressure cycling in the high-temperature (~289°C) environment. The seal has been modified to minimize dilution of the electrolyte by slow transport through the high-temperature region of the electrode. This will allow more reliable potential measurements during long term tests.

Subtask F: Mechanistic Studies (F. A. Nichols and T. F. Kassner)

The topical report entitled "Mechanistic Aspects of Stress-Corrosion Cracking of Type 304 Stainless Steel in LWR Systems" has been reviewed and revised and is currently being processed for release.

The apparatus for the Mode I versus Mode III SCC experiments has operated satisfactorily in an autoclave at 289°C and 8.3 MPa pressure. Specimens for the initial series of experiments in simulated BWR-quality water have been heat treated.

Table I. Crack Propagation Rates in Type 304 SS (Heat No. 10285) in 8 ppm O_2 Water 288°C

f (Hz)	R	K_{mx} ($MPa \cdot m^{1/2}$)	$\dot{a}$(m/s)	EPR (C/cm^2)
0	1	33-34	1.2×10^{-10}	4
2×10^{-3}	0.5	30-33	3.4×10^{-9}	
2×10^{-3}	0.6	32-33	6.6×10^{-10}	
1×10^{-1}	0.94	30-31	3.1×10^{-10}	
1×10^{-1}	0.94	31-32	1.9×10^{-10}	
0	1	32-33	2.2×10^{-10}	15
2×10^{-3}	0.5	30-32	2.8×10^{-9}	
2×10^{-3}	0.6	28-29	5.6×10^{-9}	
1×10^{-1}	0.94	30	2.1×10^{-10}	

Table II. CERT Experiments Performed in January 1983 on Type 304 Stainless Steel Specimens[a] (Heat 30956) in water with Different Oxygen, Hydrogen, and Sulfate Concentrations at 289°C and a Strain Rate of 1×10^{-6} s^{-1}

Test No.	Material EPR Value, C/cm^2	Feedwater Chemistry					Failure Time, h	Maximum Stress, MPa	Total Elong., %	Reduction in Area, %
		Oxygen, ppm	Hydrogen, ppm	$SO_4^=$, ppm	pH at 25°C	Cond., μS/cm				
49	2	0.03	0.5	1.0	4.8	8.0	119	495	43	40
50	20	0.03	0.5	1.0	4.8	8.0	115	502	42	34
51	2	0.03	0.5	10.0	3.8	71	67	383	24	25
A2	2	0.24	0	0	6.2	0.15	166	493	60	66
A3	2	0.20	0	0.1	5.7	0.8	79	402	29	37
A4	2	0.18	0	0.23	5.4	2.0	58	363	21	25

[a]Specimens were preexposed to the environment for ~20 h at 289°C before straining.

189A IDENTIFICATION NUMBER	TITLE
	Environmentally Assisted Cracking in Light Water Reactors

SUB-TASK IDENT. NO.	SUB-TASK TITLE	DATE
(A)	Leak Detection and NDE	

MILESTONE / ACTIVITY IDENT	TITLE	Start	Scheduled (∇)
A.	Review of acoustic leak detection work to date.		FY 82 O
B.	Define requirements for leak test system.		FY 82 O
C.	Design and fabricate pipe leak test system for acoustic leak characterization diagnostic experiments.	FY 82 O	FY 82 A
D.	Test leak generation system and carry out preliminary experiments [on frequency spectra (rms values) and cross-correlation techniques] to determine effect of leak geometry, pressure and temperature on leak signal.	FY 82 D	FY 83 A
E.	Complete the assessment of acoustic leak detection system capabilities.	FY 82 A	FY 83 S
F.	Assemble preliminary prototype acoustic leak detection system.	FY 83 O	FY 83 S
G.	Establish requirements for field demonstration.	FY 83 S	FY 84 S
H.	Field demonstration feasibility if established.	FY 83 J	
I.	Evaluate acoustic commercial systems as requested.	FY 82 J	
J.	Develop and test ultrasonic testing concept for special probes.	FY 82 O	FY 82 S
K.	Select special probes for further development.	FY 83 O	FY 83 J

LEGEND: ∇ SCHEDULED ▼ COMPLETED ◇ ANTICIPATED SLIPPAGE ◆ ACTUAL SLIPPAGE

189A IDENTIFICATION NUMBER

TITLE
Environmentally Assisted Cracking in Light Water Reactors

SUB-TASK IDENT. NO. (A)

SUB-TASK TITLE Leak Detection and NDE

DATE

MILESTONE / ACTIVITY IDENT	TITLE	FY 82												FY 83												FY 84											
		O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S
L.	Complete special probe evaluation for ultrasonic testing.																																				▽

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189A IDENTIFICATION NUMBER

TITLE

Environmentally Assisted Cracking in Light Water Reactors

SUB-TASK IDENT. NO. (B)

SUB-TASK TITLE: Analysis of Sensitization

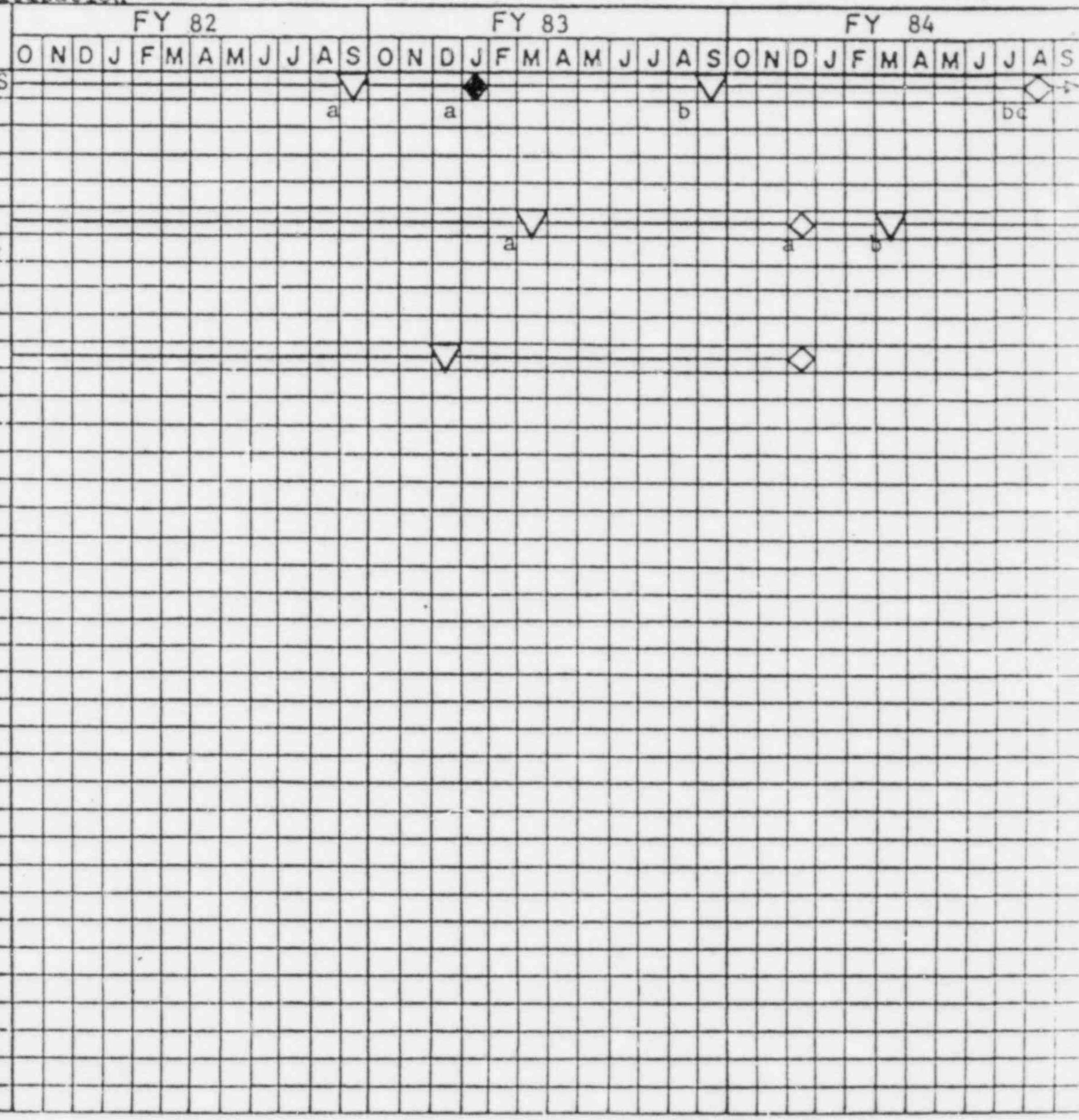
DATE

MILESTONE / ACTIVITY IDENT — TITLE

A. Effect of stress improvement remedies on LTS
- a. Welding and remedy treatments
- b. Short-term aging
- c. Long-term aging

B. Path dependence of thermomechanical histories of IGSCC susceptibility and EPR
- a. Preliminary test
- b. Long-term test

C. EPR for alternate alloys



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189A IDENTIFICATION NUMBER

TITLE
Environmentally Assisted Cracking in Light Water Reactors

SUB-TASK IDENT. NO. (C)

SUB-TASK TITLE
Crack Growth Rate Studies

DATE

MILESTONE / ACTIVITY IDENT TITLE	FY 82												FY 83												FY 84											
	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S
A. Effect of loading history															▼ a									▽ c										◇ a		
a. Comparison between high R and constant load tests																																				
b. Interaction between low R start-up and shut-down cycles and steady-state loading																																				
c. Correlation of R and frequency effects on crack growth rates with crack tip strain rates																																				
B. Effect of specimen geometry																																				
C. Piping verification tests																																				

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189A IDENTIFICATION NUMBER	TITLE	
	Environmentally Assisted Cracking in Light Water Reactors	
SUB-TASK IDENT. NO. (D)	SUB-TASK TITLE Non-environmental Corrective Actions	DATE

MILESTONE / ACTIVITY IDENT TITLE

A. Small specimen water chemistry screening tests on alternate materials
- a. Type 316NG SS
- b. Long-term very slow strain-rate tests ($\dot{\varepsilon} = 10^{-7} - 10^{-8}\ s^{-1}$)
- c. Crack-growth test on fracture mechanics specimens

B. Establish pipe test facilities and carry out initial pipe tests under alternate loading conditions

C. Analytical studies on stress redistribution
- a. Initial studies on 4-in. pipes
- b. Initial studies on large diameter pipes with IHSI
- c. Comparative studies on large and small pipes with IHSI
- d. Revise constitutive models

D. Develop stress/strain/strain-rate relations for sensitized materials

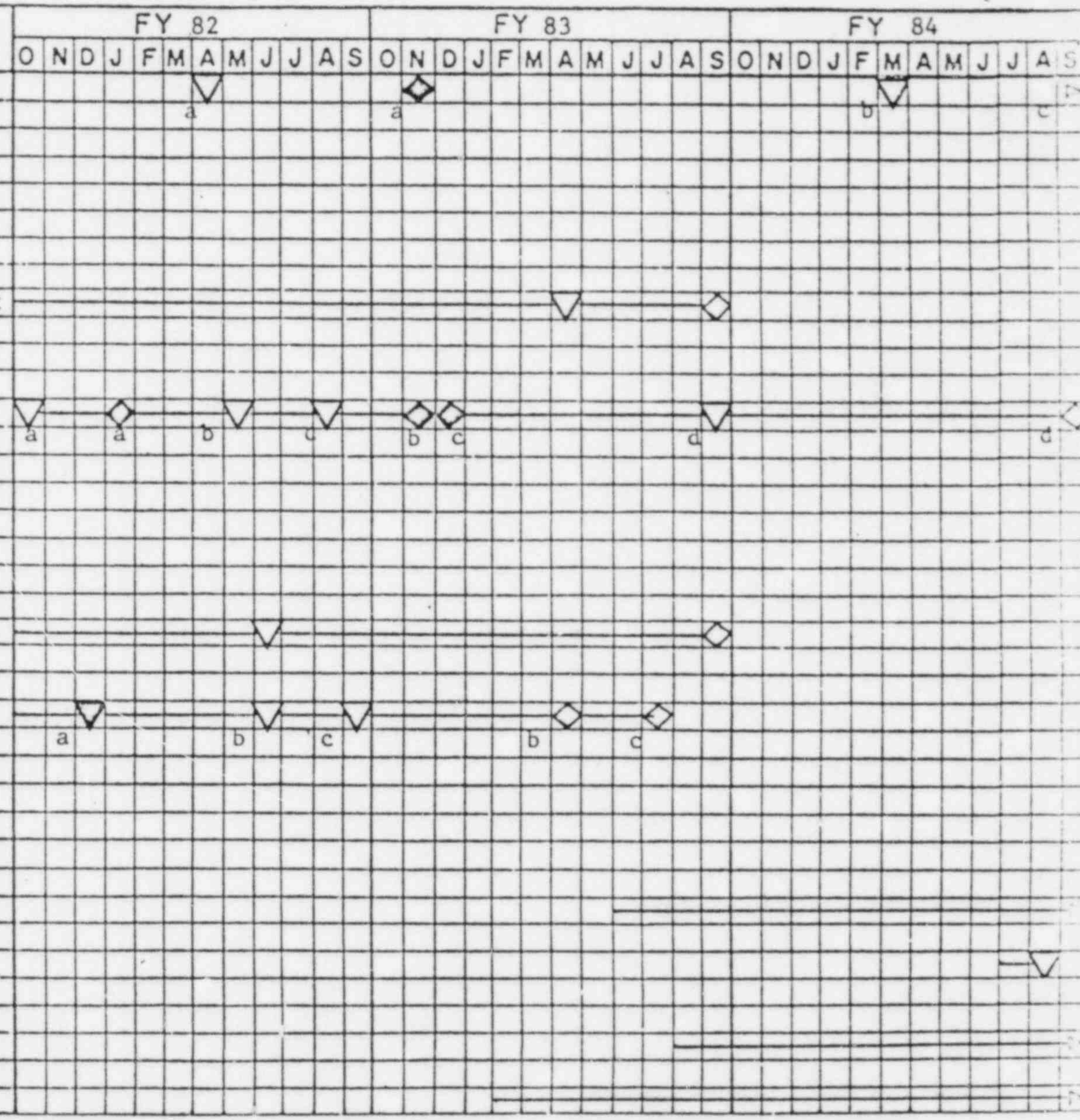
E. Benchmark tests on piping
- a. ID residual stress
- b. Strain distributions under cyclic loading
- c. Residual stress distributions after cyclic loading

F. Load history tests on smooth specimens

G. Upgrade pipe test facilities for transient testing

H. Parametric pipe tests

I. Small specimen parametric tests



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189A IDENTIFICATION NUMBER

TITLE
Environmentally Assisted Cracking in Light Water Reactors

SUB-TASK IDENT. NO.
(E)

SUB-TASK TITLE
Evaluation of Environmental Corrective Actions

DATE

MILESTONE / ACTIVITY
IDENT TITLE

A. Construct water loops for use with the third and fourth MTS Testing Systems

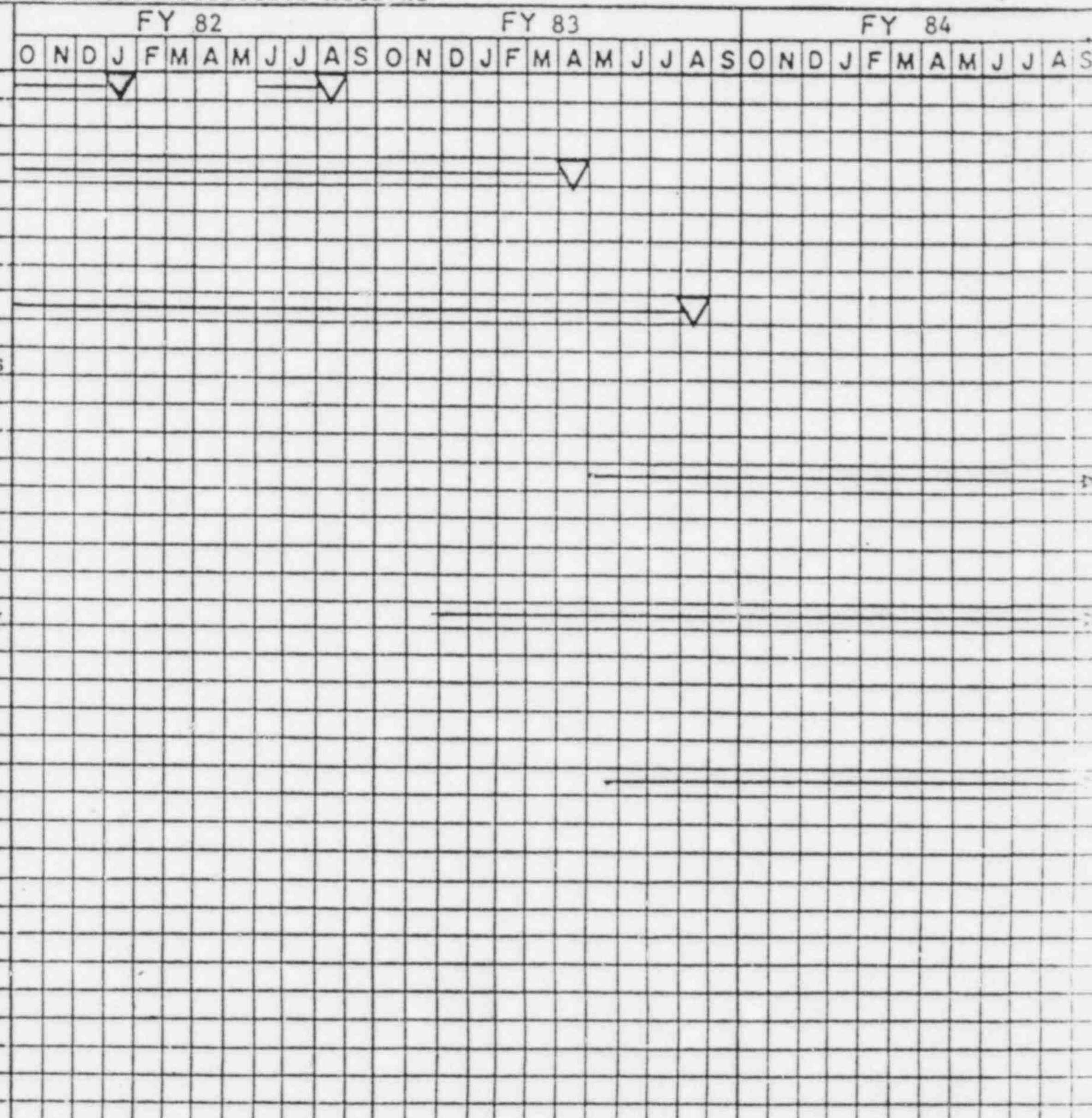
B. Perform base-line crack-growth measurements on sensitized Type 304 SS as a function of load ratio, temperature, and oxygen concentration in high-purity water

C. Investigate the effect of pH (H_2SO_4), dissolved oxygen and hydrogen, strain rate, and temperature on the IGSCC characteristics of sensitized Type 304 SS in CERT experiments

D. Determine the effect of other impurity anions (viz., Cl^-, $CO_3^=$ and $S_2O_3^=$) on the IGSCC characteristics of sensitized Type 304 SS in CERT experiments

E. Investigate the effects of coolant chemistry (viz., dissolved oxygen, hydrogen, pH, and H_2SO_4) on crack growth in Type 304 SS as a function of frequency and K_{max} at a high R value.

F. Determine the effect of water chemistry transients and flow rate on crack growth rates in sensitized Type 304 stainless steel



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189A IDENTIFICATION NUMBER

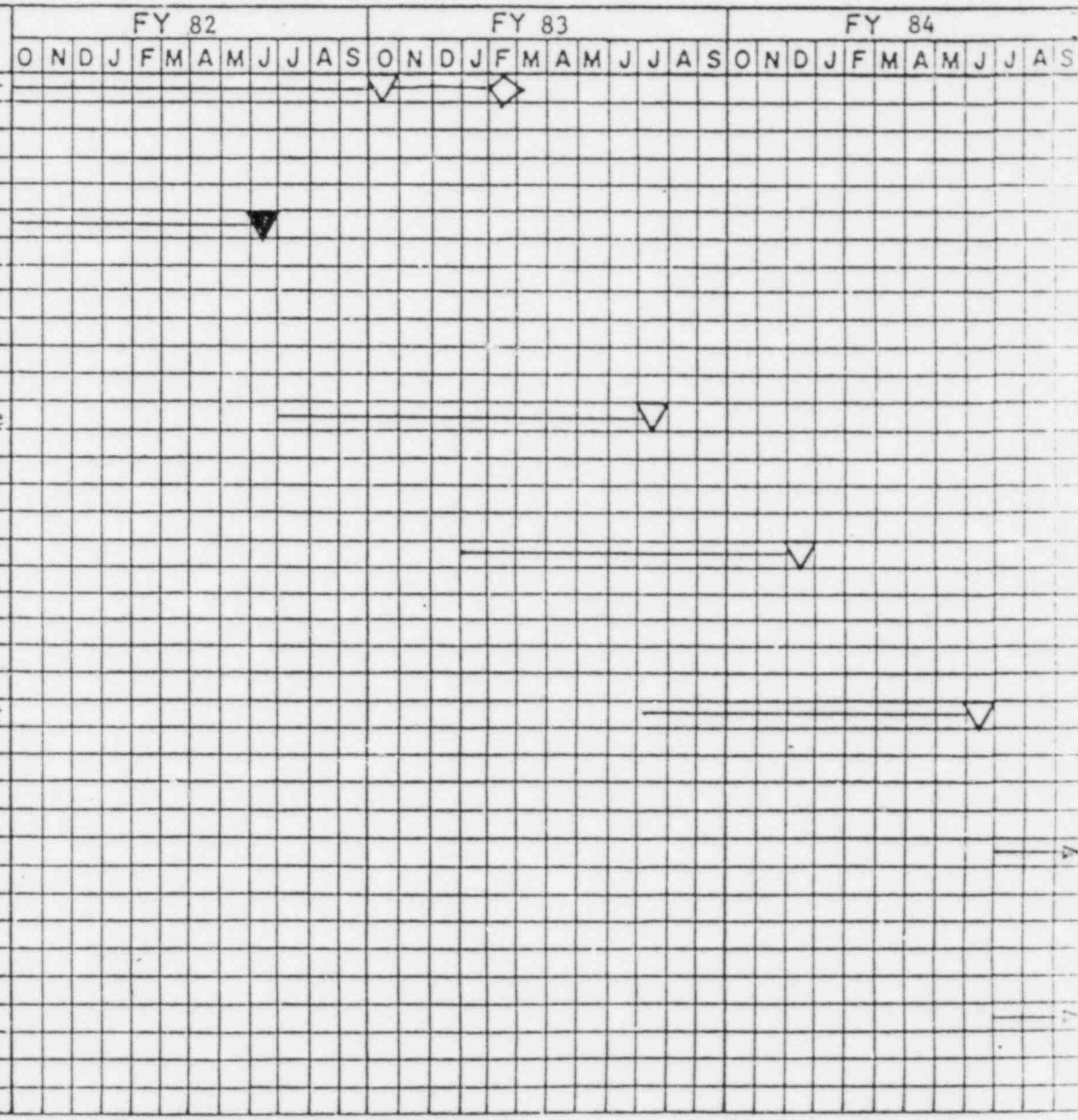
TITLE
Environmentally Assisted Cracking in Light Water Reactors

SUB-TASK IDENT. NO. (F)

SUB-TASK TITLE Mechanistic Studies

DATE

MILESTONE / ACTIVITY IDENT	TITLE
A.	Review models for various microprocesses involved in SCC of austenitic and ferritic steels in aqueous environments and prepare a topical report
B.	Assemble small refreshed autoclave systems for calibration of reference and pH electrodes, electrochemical measurements of passive film breakdown on stainless steels in simulated crack-tip solutions, and Mode I/Mode III crack-growth experiments
C.	Evaluate the performance of high-temperature Ag/AgCl reference and $OZrO_2$-Cu/Cu_2O pH electrodes for use in electrochemical measurements
D.	Construct loading system and perform Mode I and Mode III loading tests on sensitized Type 304 SS in simulated BWR-quality water at high temperature to identify the mechanism of crack growth
E.	Determine experimental parameters needed for models of crevice chemistry of Type 304 SS and correlate crack tip with bulk solution chemistry at high temperature
F.	Conduct critical experiments to identify microprocesses involved in the initiation and propagation of stress-corrosion cracks in other structural materials in simulated reactor environments
G.	Develop predictive models for crack initiation and growth in reactor materials under different environmental conditions



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