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REACTOR PRIMARY COOLANT SYSTEM PIPE RUPTURE STUDY METHOD FOR DETECTION OF SENSITIZATION IN STAINLESS STEEL PROGRESS REPORT NO. 42

OCTOBER—DECEMBER 1977

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METHOD FOR DETECTION OF SENSITIZATION IN STAINLESS STEEL
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ABSTRACT

A program is in progress to develop a quantitative method (EPR) for nondestructively measuring the degree of sensitization in Type-304 and -304L stainless steels. The technique has been extended to characterize weld heat-affected zones and to correlate degree of sensitization with intergranular stress-corrosion cracking resistance. Current studies are directed toward establishing procedures for, and qualifying, a technique to obtain EPR measurements in-situ on reactor components in the field.

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I. INTRODUCTION

The Reactor Primary Coolant System Pipe Rupture Study is being conducted for the Metallurgy and Materials Research Branch of Division of Reactor Safety Research, USNRC. This Progress Report No. 42 documents work done in the period October 1, 1977 to December 31, 1977. The over-all objective of the program is to improve the reliability of reactor system piping by increasing current knowledge of failure-causing mechanisms and by enhancing capability for design evaluation and analysis.

Toward the attainment of this objective, work was continued on the following basis:

Task G - Effect of Sensitization on Type-304 Stainless Steel
Task K - Effect of BWR Environment

All testing under Task K has been completed; no further progress will be reported. A topical report* was issued this period. A second report** is nearly completed. These two documents will summarize the results developed during Task K, which will terminate the NRC-sponsored portion of the project.

Most of the Task G progress for this period was reported in a topical report*** in process. Consequently this report will only cover recent progress not included in GEAP-12697.

The specific objectives of Task G for fiscal year 1978 include:

1. Investigation of alternate methods for obtaining EPR measurements of components in the field. These include trepanning, small cell in-situ measurements in a drilled hole, "boat" sampling and subsequent laboratory measurement after heat treating, and pipe outside spot checking after localized heat treatment.

*D. A. Hale, et al., "Low Cycle Fatigue Evaluation of Primary Piping Materials in a BWR Environment," September 1977 (GEAP-20244).

**D. A. Hale, J. Yuen, and T. L. Gerber, "Fatigue Crack Growth in Piping and RPV Steels in Simulated BWR Water," to be issued (GEAP-24098).

***W. L. Clarke and V. M. Romero, "Detection of Sensitization in Stainless Steel: II. EPR Method for Nondestructive Field Tests," to be issued (GEAP-12697).

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2. Resolution of technological problems encountered during fiscal year 1977 investigation, i.e., grain size effects, surface and structural effects such as cold work produced by grinding, martensite formation, etc.

3. Fabricate a cell capable of obtaining EPR measurements on the inside of a pipe during the construction phase of reactor installation.

4. Develop a larger data base (more heats and product forms) to provide more confidence in establishing "safe limits" of sensitization for welded components, in terms of stress-corrosion resistance under the actual environmental and stress conditions of concern.

5. Conduct a "round robin" test in conjunction with ASTM Committee G1.08 to initiate adoption procedures for acceptance of the EPR technique as an ASTM standard.

QUARTERLY REPORTS

GEAP-4911	Reactor Primary Coolant System Rupture Study Quarterly Progress Report No. 1, April - June 1965
GEAP-4964	No. 2, July - August 1965
GEAP-5082	No. 3, October - December 1965
GEAP-5147	No. 4, January - March 1966
GEAP-5192	No. 5, April - June 1966
GEAP-5279	No. 6, July - September 1966
GEAP-5427	No. 7, October - December 1966
GEAP-5474	No. 8, January - March 1967
GEAP-5512	No. 9, April - June 1967
GEAP-5554	No. 10, July - September 1967
GEAP-5587	No. 11, October - December 1967
GEAP-5637	No. 12, January - March 1968
GEAP-5680	No. 13, April - June 1968
GEAP-5716	No. 14, July - September 1968
GEAP-5770	No. 15, October - December 1968
GEAP-10024	No. 16, January - March 1969
GEAP-10072	No. 17, April - June 1969
GEAP-10120	No. 18, July - September 1969
GEAP-10143	No. 19, October - December 1969
GEAP-11069	No. 20, January - March 1970
GEAP-10207	No. 21, April - June 1970
GEAP-10207-22	No. 22, July - September 1970
GEAP-10207-23	No. 23, October - December 1970
GEAP-10207-24	No. 24, January - June 1971
GEAP-10207-25	No. 25, July - September 1971
GEAP-10207-26	No. 26, October 1971 - March 1972
GEAP-10207-27	No. 27, April - June 1972

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GEAP-10207-28	No. 28, July - December 1972
GEAP-10207-29	No. 29, January - June 1973
GEAP-10207-30	No. 30, July - December 1973
GEAP-10207-31	No. 31, January - June 1974
GEAP-10207-32	No. 32, July - December 1974
GEAP-10207-33	No. 33, January - June 1975
GEAP-10207-34	No. 34, July - December 1975
GEAP-10207-35	No. 35, January - March 1976
GEAP-10207-36	No. 36, April - June 1976
GEAP-10207-37	No. 37, July - September 1976
GEAP-10207-38	No. 38, October - December 1976
GEAP-10207-39	No. 39, January - March 1977
GEAP-10207-40	No. 40, April - June 1977
GEAP-10207-41	No. 41, July - September 1977

TOPICAL REPORTS

GEAP-4445	A Survey of Water Reactor Primary System Conditions Pertinent to the Study of Pipe Rupture, January 1965
GEAP-4446	A Review of Fracture Modes as Related to Reactor Primary Coolant Pipe Rupture, May 1964
GEAP-4474	Experimental and Analytical Program Recommendations - Reactor Pipe Rupture Study, January 1965
GEAP-4574	Survey of Piping Failures for the Reactor Primary Coolant Pipe Rupture Study, May 1964
GEAP-4578	Mechanical Design Considerations in Primary Nuclear Piping, March 1964
GEAP-4678	Fracture Mechanics and the Stability of Engineering Structures, September 1965
GEAP-5014	Fracture Toughness of Some Engineering Alloys, March 1966
GEAP-5410	Low-Cycle Fatigue Strength Reduction in Notched Flat Plates, January 1967
GEAP-5471	PAPA - Structural Analysis of Plates and Shells Using Trapezoidal and Triangular Plate Elements, March 1967
GEAP-5553	PAPA - The Digital Computer Program for the Static Analysis of Structures Made Up of Plate and Panel Elements, October 1967
GEAP-5557	Stress Intensity for a Circumferential Through-Wall Crack in a Straight Pipe, January 1968
GEAP-5607	Fatigue Crack Growth in Nuclear Reactor Piping Steels, March 1968
T&AM-677	A Three-Dimensional Photoelastic Study of Stress Near Cracks in Shell Structures, University of Illinois, March 1968

GEAP-5620	Failure Behavior in ASTMA 106-B Pipes Containing Axial Through-Wall Flaws, April 1968
GEAP-5622	A Failure Diagram for Axially Flawed Pipes, April 1968
GEAP-5653	Status of Pipe Rupture Study at General Electric - II, July 1968
GEAP-5614	Notched High-Strain Fatigue Behavior of Three Low-Strength Structural Steels, September 1968
GEAP-5724	BWR Primary Piping Stresses for System Reliability Analysis, September 1968
GEAP-5726	Influence of Stress/Strain Concentration and Mean Stress on the Low-cycle Fatigue Behavior of Three Structural Steels at Room Temperature, September 1968
GEAP-10007	Restart for the PAPA Program, March 1969
GEAP-10090	The 550°F Notched High-Strain Fatigue Behavior of Three Low-Strength Structual Steels, August 1969
GEAP-10135	Low-Cycle Fatigue of Prototype Piping, January 1970
GEAP-10140	The Effect of Cyclic Strain Aging on the Embrittlement of a Plain Carbon Steel Plate, July 1970
GEAP-10170	The Effect of Stress Concentration on the Low-Cycle Fatigue of Three Low-Strength Structual Steels at Room Temperature and 500°F, March 1970
GEAP-10181	Low-Cycle, Strain-Controlled Fatigue Crack Propagation in Nuclear Piping Steels, May 1970
GEAP-10205	Status of Pipe Rupture Study at General Electric Company, Part III, June 1970
GEAP-10236	Failure Behavior of Flawed Carbon Steel Pipes and Fittings, October 1970
GEAP-10452	Estimating Pipe Reliability by the Distribution of Time-to-Damage Method, March 1972
GEAP-10763	Low-Cycle Fatigue of Prototype Pipe Components, January 1973
GEAP-20662	Effect of Constraint and Loading Mode on Low-Cycle Fatigue Crack Initiation - Comparison with Code Design Rules, October 1974
GEAP-20615	Estimating the Relative Probability of Piping Severance by Fault Cause
GEAP-21382	Detection of Sensitization in Stainless Steel Using Electrochemical Techniques, August 1976
GEAP-20244	Low-Cycle Fatigue Evaluation of Primary Piping Materials in a BWR Environment, September 1977

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2. RESULTS AND DISCUSSION

2.1 PIPE INSIDE CELL FABRICATION

Fabrication drawings were prepared and a pneumatic actuator obtained to assemble an EPR cell for inside pipe measurements. This cell was to be similar in size and dimensions to the outside pipe cell discussed in detail in GEAP-12697. However, new miniaturized EPR cells have been designed and fabricated (discussed next) which are superior to the larger, early cell designs. Consequently, the larger pneumatic actuated cell concept has been abandoned in preference to the improved miniature cell design.

2.2 MINIATURIZED EPR CELL DEVELOPMENT

Two miniaturized EPR cells were designed and fabricated this period (Figures 1 and 2). Both cell designs utilize Pt wire counter electrodes, and porous ceramic plugged Teflon capillary tubing for reference electrode salt bridges. The cells shown in Figure 1 were fabricated from Plexiglas, and are sealed to the working electrode using ethylene-propylene gaskets. The ends of these cells are machined to match the curvature on the inside and outside of various diameter pipes.

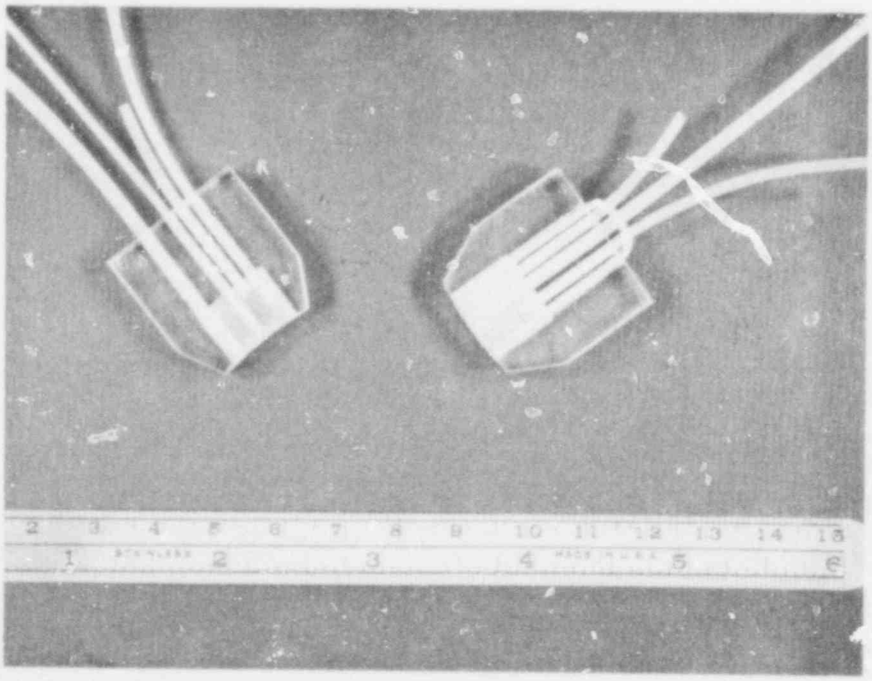

FIGURE 1. MINIATURIZED PLEXIGLAS CELLS FOR OBTAINING NONDESTRUCTIVE EPR MEASUREMENTS ON PIPING (o.d. cell left, i.d., cell at right)

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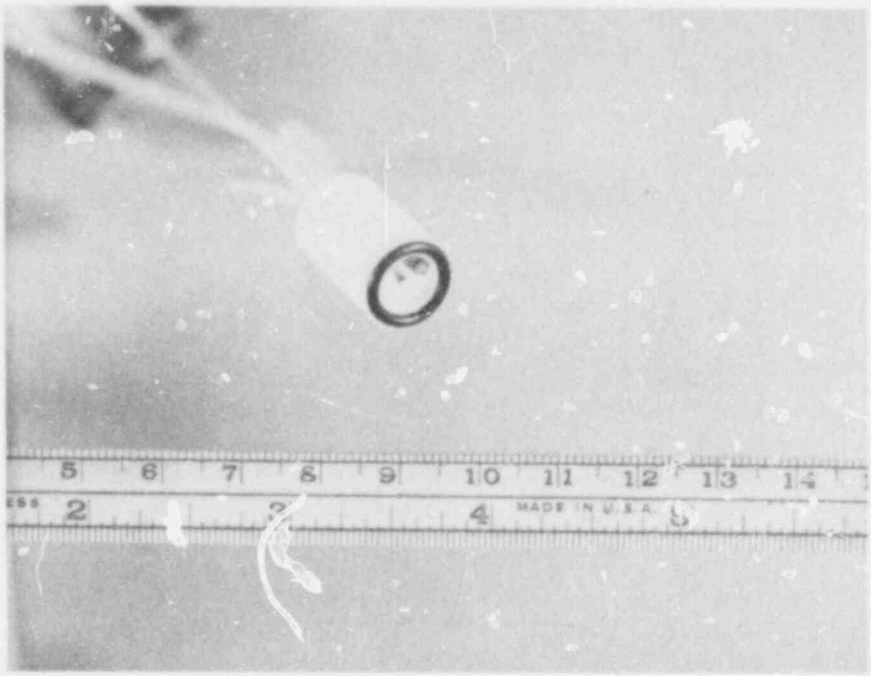

FIGURE 2. MINIATURIZED TEFLON CELL FOR NONDESTRUCTIVE EPR MEASUREMENT. NOTE "O" RING SEAL ON END, Pt COUNTER ELECTRODE IN CENTER OF CAVITY AND POROUS CERAMIC PLUGGED CAPILLARY REFERENCE ELECTRODE BRIDGE (Top of Cavity)

The second cell (Figure 2) is round in geometry, and was fabricated from Teflon bar stock. The cell contains an ethylene-propylene "O" ring for a seal and the same counter and reference electrode arrangement described for the Plexiglas cells. The round Teflon cell (1.27-cm diam x 2-cm long) can also be used on pipe outside and inside diameters (Figures 3 and 4, respectively), and is more useful for attaching to flat components. Preliminary measurements to qualify the miniature cells were obtained by conducting EPR tests on the faces of mounted samples which were previously tested using the conventional laboratory beaker technique. These data are given in Table 1, where good agreement between the two measurement methods is indicated using a variety of sensitized Type-304 stainless steel samples.

2.3 ALTERNATE FIELD TEST METHODS

2.3.1 Drilled Hole Technique

An EPR assessment of degree of sensitization was made in-situ on a segment of furnace sensitized (620°C/40 h/FC) Type-304 stainless steel pipe (heat M 7772), by measurement

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TABLE 1. EPR Measurements Comparing Miniaturized Field Cell to Conventional Laboratory Method
(0.5 M H_2SO_4 + 0.01 M KSCN at 30°C)
(Type-304 Stainless Steel)

Heat	Heat Treatment	EPR (Pa, C/cm²) Conventional Beaker Test	Field Cell Test
78500	1300°F/1 h/FC	67.7	37.8
M7772	Welded HAZ	0.4	0.7
M7772	Welded HAZ	0.4	1.9
CR&D	Cooling Rate Study	1.8	1.9
CR&D	Annealed	0.0	0.1
834264	Welded HAZ	0.5	0.4
834264	Welded HAZ	1.1	1.6

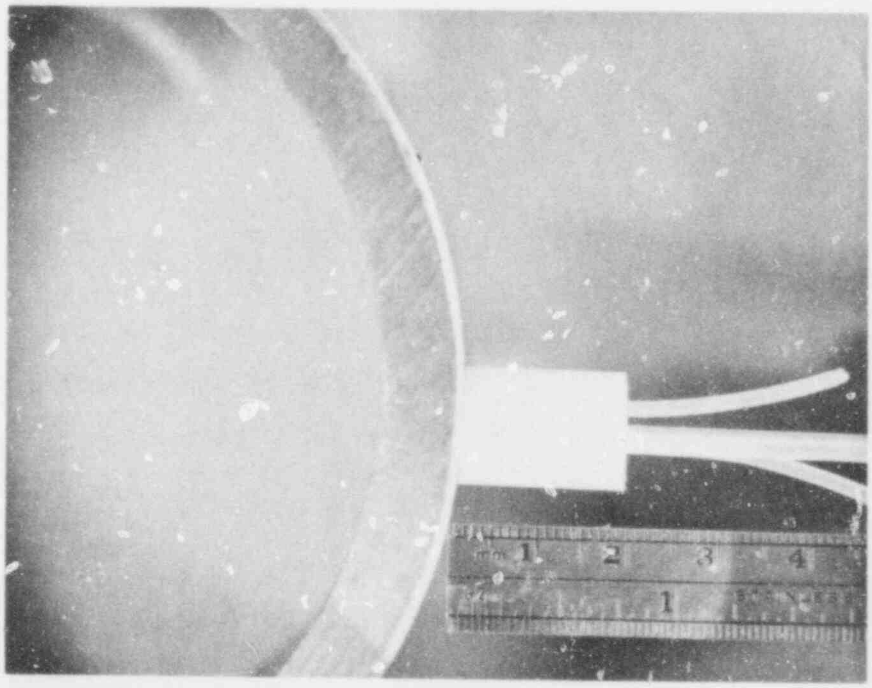

FIGURE 3. TEFLON CELL SHOWN ATTACHED TO OUTSIDE OF 4-in. PIPE

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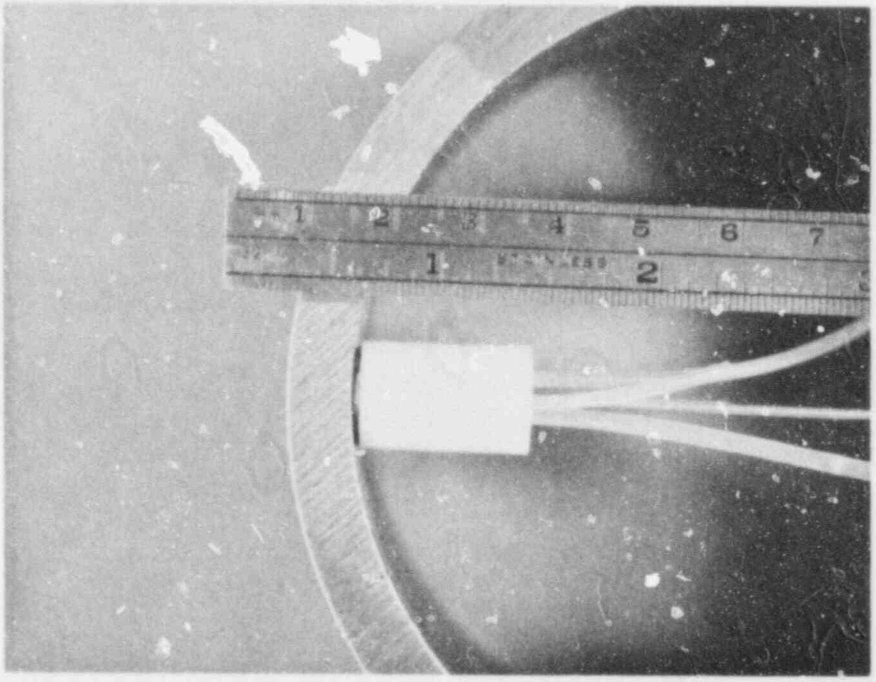

FIGURE 4. TEFLON CELL SHOWN ATTACHED TO INSIDE OF 4-in. PIPE

at the base of a 1.27-cm (0.5 in.) diameter hole bored 6.3 mm (0.250 in.) into the pipe wall. The side-walls of the bored hole were masked using stop-off lacquer, so that only the base of the hole was analyzed. The hole was then filled with the 0.5 M H_2SO_4 + 0.01 M KSCN electrolyte and the counter and reference electrode capillaries inserted into the hole. The EPR measured Pa values obtained were 35.6 and 40.2 (repolished base) C/cm^2, which is consistent with the 22 C/cm^2 measured for this heat after a 24-hour heat treatment at 620°C, using the conventional laboratory technique. The next measurement was obtained by inserting the circular Teflon cell into the hole, using the "O" ring to seal off a portion of the hole base. The EPR value obtained here was 47.3 C/cm^2, which is in good agreement with the in-situ measurement. Finally, the above small cell was attached to the inside surface of the pipe (refer to Figure 4), where a value of 39.0 C/cm^2 was measured. A representative photomicrograph is shown in Figure 5 which typifies the structures observed within the drilled hole, and using the small cell, after all of the EPR measurements on the furnace sensitized pipe.

Therefore, it appears that the drilled hole with or without the miniaturized cell is a viable in-situ measurement technique, providing the area of interest can be analyzed. Additional

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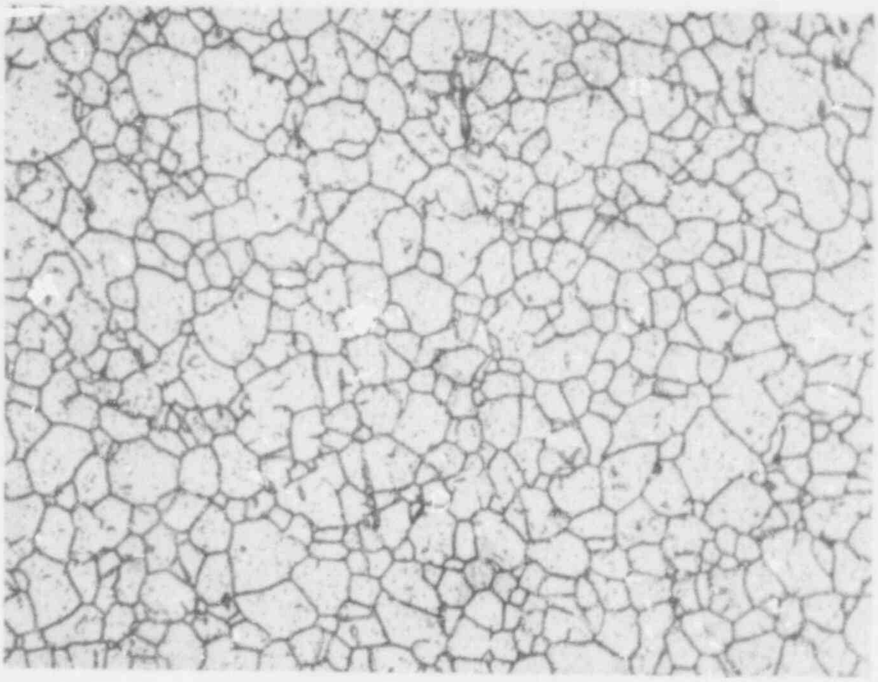

FIGURE 5. MICROSTRUCTURE ETCHED IN FURNACE SENSITIZED TYPE-304 PIPE BY EPR MEASUREMENT USING MINIATURIZED TEFLON CELL

work will concentrate on measurement of weld HAZ in drilled holes at depths where satisfactory correlation with pipe inside values are obtained.

2.3.2 Surface Spot-Heating Technique

One alternate field measurement technique under study consists of spot (~ 2.5 cm diam) heating the surface of a pipe to temperatures and times simulating welding practices. Thus, outside surface EPR measurements could be taken using the miniaturized cells and the portable polarization system. The occasional heats of Type-304 stainless steel which are exceptionally susceptible to sensitization after thermal treatments could then be identified. These "bad" heats are presumably more susceptible to intergranular stress-corrosion cracking (IGSCC) after welding than the average. Hence, these heats could be tagged for more detailed and/or more frequent inspection during normal reactor service. The spot heating method must only affect a shallow area on the surface of the component, so that the inside (exposed to coolant) does not become sensitized [temperature not to exceed 425°C (800°F)].

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A commercially available spot heater was obtained for demonstration purposes. The particular instrument obtained was a poor demonstration model, although, with proper insulation, we managed to achieve temperatures up to 600°C (1112°F) for 10 minutes to the surface of a 10.16-cm (4-in.) pipe section, The corresponding temperatures on the inside of the pipe directly beneath the heated spot reached 350°C (662°F) as a maximum.

A better technique for spot heating appears to be the use of a cone-directed heat from a welding torch. The time and temperature could be controlled by a thermocouple connected to a programmable attachment on the welding power source. These attachments are relatively simple and are adaptable to modern power sources which are probably available at more reactor sites. This latter technique will be investigated further next period.

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