

# GENERAL ELECTRIC

NUCLEAR POWER  
SYSTEMS DIVISION

GENERAL ELECTRIC COMPANY, 175 CURTNER AVE., SAN JOSE, CALIFORNIA 95125  
MC 682, (408) 925-5040

MFN-002-80

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

Attention: Mr. Stephen H. Hanauer, Director  
Unresolved Safety Issues Program

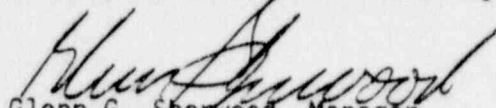
Gentlemen:

SUBJECT: TECHNICAL REPORT ON MATERIAL SELECTION AND PROCESSING  
GUIDELINES FOR BWR COOLANT PRESSURE BOUNDARY PIPING",  
NUREG-0313, RFV. 1

General Electric comments on the subject document, noticed in the November 16, 1979 issue of the Federal Register, are enclosed.

Please note that the technical comments are contained on the first six pages; the remainder of the material is enclosed for back-up. This material consists of Appendices A through H and Attachments A, B, and C. The back-up material is very important in justifying GE comments and should be distributed to all reviewers.

If you have any questions on the enclosures, please contact Mr. R. L. Gridley at (408) 925-3732 of my staff.

  
Glenn G. Sherwood, Manager  
Safety & Licensing Operation

GGs:rm/104Q

Enclosures

cc: Mr. Darrel G. Eisenhut, Acting Director  
Division of Operating Reactors  
Office of Nuclear Reactor Regulation

Mr. L. S. Gifford, GE-Bethesda

X601  
2/2  
END TO:  
FILES  
NRC PDR  
S HANAUER  
C CHENG  
R GAMBLE  
R TUROVLI  
A TABOADA  
OTHERS RECEIVE  
LTR  
ADD:  
D EISENHUT  
ON A/COCK  
800110  
375  
110  
110

90014001

## INTRODUCTION

General Electric feels that expeditious implementation of technological advances is important to the future of nuclear power, which in turn is essential to meeting the energy needs of this nation. While selective inspection of components and systems which have been identified as subject to Stress Corrosion Cracking is beneficial for the efficient operation of nuclear reactors, the benefits of inspection must be weighed against the hazards of exposing non-destructive testing personnel to significant levels of radiation.

The attached comments are organized in the following manner: the NUREG 0313 page and paragraph are indicated, followed by suggested wording changes. A brief statement of GE's rationale for the suggested change follows with reference to an appendix where a more complete discussion of GE concerns is given plus references, papers and other supporting documentation.

90014002

GE COMMENTS

NUREG 0313

Page &  
Paragraph

Page 1 & Title

Insert "Stainless Steel" after "BWR Coolant" and before "Pressure Boundary Piping".

200°F Minimum Limit for Inspection of Piping

Page 1  
Paragraph 2

Change to read, "The guidelines. . . have been modified to include ASME Code Class 2 piping and to exclude pipe whose normal service temperature is at or below 200°F.

NUREG 0531, page 4.6, 4.4.4 recognizes that "increasing temperature in BWR environments. . . increases the susceptibility to IGSCC" Reg. Guide 1.44 in paragraph C.4 a recognized that stress corrosion was not a concern below 200°F. Neither NRC pipe crack studies nor GE pipe crack reviews have documented a stress corrosion problem in BWR piping at or below 200°F. Selective inspection of identified problem areas is far more useful in detecting cracks and reduces unnecessary radiation exposure to personnel.  
See Appendix A.

Use of Cast Austenitic Stainless Steel

Page 7  
Paragraph II.A.1

Substitute "Cast Austenitic Stainless Steels with  $\geq$  5% Ferrite "for" Type CF3 Cast Stainless Steel

This material has never cracked by IGSCC in a BWR pressure boundary. Service and laboratory experience have been excellent. Classing cast austenitic stainless steels with 304 will result in increased inspection and resultant unwarranted radiation exposure to NDT Inspectors. Omission of some grades of this material would improperly emphasize augmented inspection where it is less valuable.  
See Appendix B.

90014003

Nitrogen in Austenitic Stainless Steels

Page 7  
Footnote

Change "These materials have . . . nitrogen (0.1% max.) to  
"These materials have . . . nitrogen (0.16% max.).

The use of slightly higher nitrogen contents than are common in normal practice is desirable because nitrogen has been identified as retarding sensitization and reducing crack growth rates in austenitic stainless steels. Slightly higher nitrogen contents are known to improve mechanical properties and compensate for low carbon levels in 304L and 316L grades. ASTM has accepted the "LN" designation and ASTM specifications for these materials are being issued. See Appendix C.

90014004



Page 7  
Section A-1

Substitute "Austenitic stainless steel weld metal with  $\geq$  5% ferrite" for "Type 308L stainless steel weld metal with at least 5% ferrite content."

The majority of field welds are 308 weld metal. Other similar alloys have also been used. The high ferrite content has been responsible for an excellent service history: No stress corrosion cracking has been observed to originate piping welds of these materials in the field. Cracks have been observed to arrest in 308 weld metal.

Conforming Processes

Page 7  
Paragraph II A.2

Add "Heat Sink Welding and Field Corrosion Resistant Cladding are considered to be conforming processes."

Page 10  
Paragraph 4

Change to read, "Heat Sink Welding has been evaluated and accepted by the NRC."

Heat Sink Welding (HSW) has performed very well in the Pipe Test Laboratory. HSW produces compressive residual stresses on the pipe I.D. surface and reduces sensitization in the weld HAZ. Field Corrosion Resistant Cladding (CRC) has also performed well in laboratory tests due to the stress corrosion resistance of the clad material overlaid on the heat affected zones of butt welds. G.E. considers that these processes are qualified for specific applications in BWR piping. See Appendices D and E.

90014005

The Electro Potentiokinetic Reactivation Test

Page 9  
Section B  
Paragraph 2

Instead of "The Electrochemical Potentiokinetic Reactivation (EPR) test has not been formally evaluated and accepted by the NRC", substitute "The Electrochemical Potentiokinetic Reactivation (EPR) test has been evaluated and accepted and is recognized as an acceptable means of screening materials for sensitization."

Under NRC sponsorship, the EPR test has been extensively tested in comparison with ASTM A-262 practices A and E. This test is superior to ASTM A-262 practices A and E in detecting sensitization in steels. EPR results have successfully predicted IGSCC susceptibility. A large number of other organizations have accepted and use this test. ASTM has recognized and accepted EPR, and a specification for this procedure is in progress. See Appendix F.

Field Corrosion Resistant Cladding for Repairs

Page 10  
Paragraph 3

Change to read "which may become sensitized as a result of a butt welding process."

This modification will allow field CRC use in BWR's. Although field CRC may result in a slight degree of sensitization in the region at the edge of the cladding, field CRC has shown a factor of improvement of 7 in severe pipe tests. Since cladding is a welding process, this paragraph as it stands can be interpreted as a prohibition of field CRC in contradiction with Section IIC which allows field CRC for repairs and in plants already under construction. See Appendix D and E.

90014006

Service Sensitive Lines

Page 11                      G.E.'s position is that Recirculation Risers and Recirculation  
Section B                    Inlet lines at safe ends where crevices are formed by the  
Paragraph 2                   welded thermal sleeve attachments should be subject to  
                                 augmented inspection proposed and approved on a case by  
                                 case basis rather than designated and "service sensitive".

Field cracking of recirculation risers and safe ends has not developed a repetitive pattern. Numerous inspections in the United States have not identified additional cracks. See Appendix G.

Applicable Code Requirements

Page 14 & 15                Add, "except for older plants where earlier code requirements  
Paragraph 1 B                are in effect."

There exists an apparent contradiction between NUREG 0313 and previously expressed NRC Policy. The NRC's policy on the effective application of the ASME Code has been, as of 10/19/79, that sections of the Code more recent than 1975 apply to plants with docket dates more recent than July 1, 1978. Older plants would be subject to the S75 code, but not to newer sections unless the utility elects to do so. The NUREG position on this matter should be clarified to make it consistent with this position.

See Appendix H.

90014007

Selection of Welds Considering Seismic and Fatigue Loads

Page 15

Omit this paragraph.

Paragraph 2b & c

Paragraph 3a

It can be demonstrated that inspection of welds and systems is more effective where the likelihood of stress corrosion cracking is higher due to factors such as high sustained stress and sensitization. Inspection of welds where significant seismic or fatigue loads have been postulated for hypothetical seismic events and where other factors which ordinarily are associated with cracking are absent seems of limited value. Stress corrosion is known to occur when associated with sustained stresses: isolated high stress transients are not a concern in this area.

General Comment to Section III

General Electric considers that the IGSCC stress rule approach, which it has used to describe cracking patterns, should be employed in selecting weld sites for additional in-service inspection. As documented in Attachment C (NEDO 23684), the stress rule technique does demonstrate acceptable field failure correlation. Accordingly, all NRC issues relating to the IGSCC stress rules should be identified and settled such that the next modification of NUREG 0313 can endorse these techniques as alternative means of selecting weld locations for augmented inspection upon case-by-case NRC approval.

90014008

APPENDIX A200°F Minimum Limit for Inspection of Piping

All piping cracks to date where service conditions are documented have occurred at temperatures well above the 200°F limit. Temperature plays a significant role in stress corrosion in the BWR environment; NUREG 0531, pages 4.6, 4.4.4 recognizes that "increasing temperature in BWR environment. . . increases the susceptibility to IGSCC". In addition, Reg. Guide 1.44 recognized that temperature plays a significant role in stress corrosion. This document requires control of materials except where "material exposed to reactor coolant which has a controlled concentration of less than 0.10 ppm dissolved oxygen at all temperatures above 200°F during normal operations".

G E. Laboratory data, attached demonstrates the relationship between temperature and the occurrence of stress corrosion. No stress corrosion has been found in welded stainless steel in a BWR environment at or below 200°F.

No low temperature stress corrosion problem in a BWR environment has been identified to date, either in NRC documentation or in G.E. reports.

The benefits of increased in-service inspection - improved crack detection rates, reduced risk of leakage, are dissipated if increased in-service inspection is applied to lines where experience has shown cracking to be highly improbable. The risk of increased radiation dose rate to NDT inspectors and the economic losses to utilities and their customers due to decreased availability must be balanced against possible advantages. Due to the lack of an identifiable problem, no such advantages are apparent in this case.

Reference: "BWR Coolant Oxygen Control" by R.L. Cowan, J.C. Elliott and O.H. Johanneson, NEDO-23631 77, NED106 Class I, June 1977. (portions attached).

90014009

POOR ORIGINAL

## 2.4 EFFECT OF TEMPERATURE

## 2.4.1 Stainless Steel

One test program has addressed the effect of temperature on the intergranular stress-corrosion response of sensitized stainless steel in high purity water.<sup>18</sup> Sensitized samples (1085°F/20h/furnace cooled) of wrought Type-304 were tested under constant extension rate (0.03 mils/min) at six temperatures in air-saturated high purity water. The results of these tests are shown in Table 3-3. From these results, one would conclude that stress-corrosion cracking could initiate during a stress event at temperatures as low as 250°F and possibly as low as 200°F. Confirming data on the effect of temperatures are shown in Figure 3-4 produced in an accelerated test under constant load conditions in oxygenated high purity water.

Table 3-3  
RESULTS OF CONSTANT DISPLACEMENT RATE TEST  
ON SENSITIZED STAINLESS STEEL<sup>18</sup>

Test Temperature (°F)	Mean Stress (ksi)	RA (%)	Failure Time (hr)	IGSCC by Fractography
200	76.5	76.5	120	No*
225	73.0	73.0	159	No*
250	71.4	53.4	116	Yes
300	69.0	53.4	118	Yes
350	66.0	27.8	79	Yes
550	39.5	27.1	74	Yes

## Test Conditions

- Samples from 6-in. Schedule 80 Type-304 Seamless Pipe (Heat No. 27368).
- Displacement Rate = 0.03 mils/hr.
- High purity, air-saturated water (~6 ppm O<sub>2</sub>).
- Samples examined by metallography and scanning electron microscopy (SEM).

\* Grain separation in one location of fracture surface, by scanning electron microscopy (SEM).

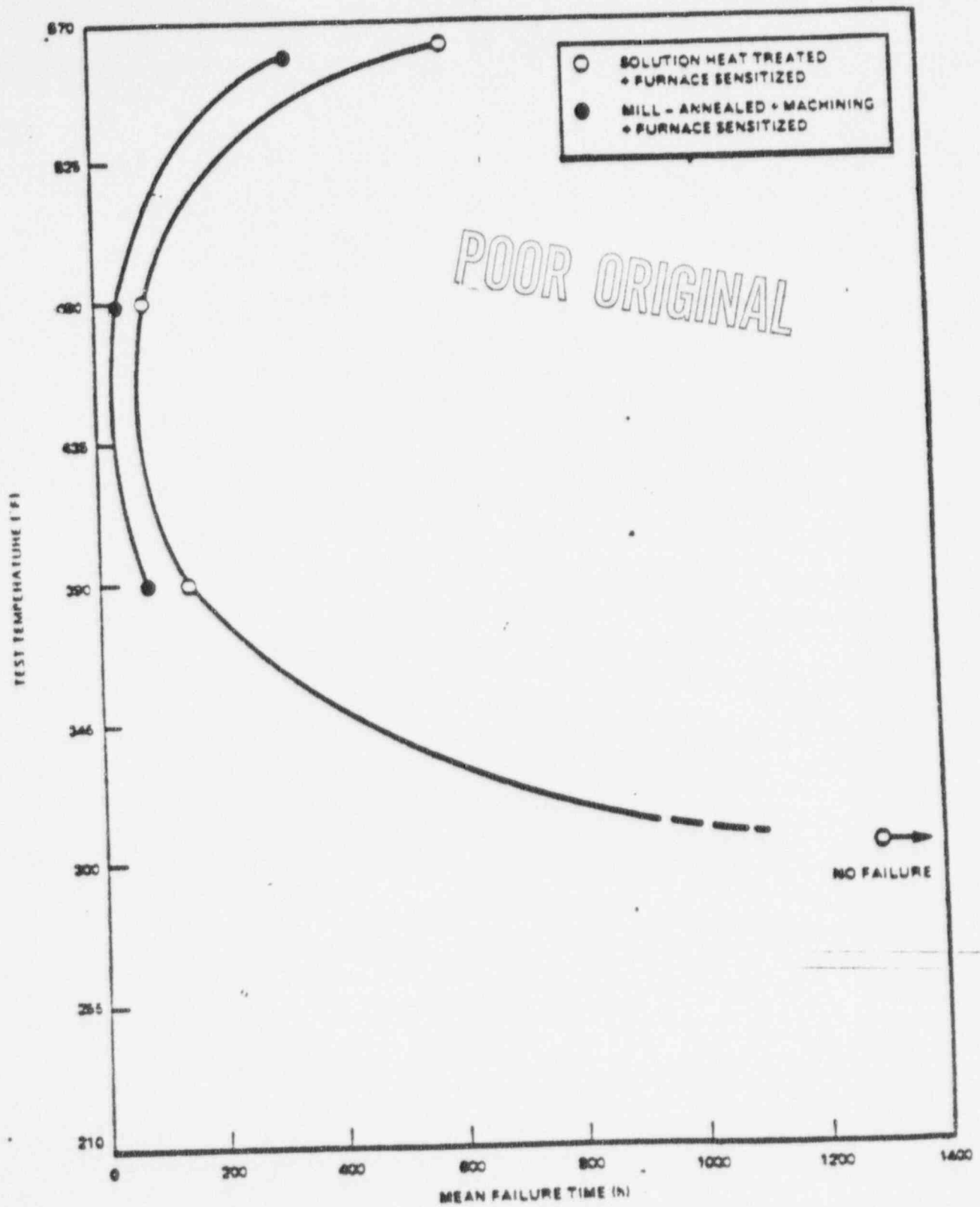


Figure 3-4. Effect of Test Temperature on Mean Failure Time to IGSCC of Sensitized Type-304 Stainless Steel in Oxygenated High Temperature Water<sup>10</sup>



Use of Cast Austenitic Stainless Steel with  $> 5\%$  Ferrite

The NUREG -0313 list of acceptable materials for IGSCC resistance, paragraph II- A-1, should include cast Austenitic stainless steel with  $\geq 5\%$  ferrite. Since the purpose of the document is to reduce the incidence of IGSCC occurrences in BWR piping, it is not clear why the NRC would choose to prohibit materials that have never cracked by IGSCC in a BWR pressure boundary. The only IGSCC incident of Cast Austenitic Stainless Steel with  $\geq 5\%$  ferrite cracking, General Electric is aware of occurred in the Dresden-2 Jet Pump transition casting. This failure was attributed to improper processing that resulted in surface carburization; surface carburization can occur in CF3 or any cast austenitic stainless steel. Surface carburization is avoided by proper process control.

General Electric performed a large test matrix on Type CF3 and CF8 castings utilizing constant extension rate tests in 550°F 8 ppm oxygenated water. The matrix shown below included high carbon and low ferrite material. All failures were ductile (no stress corrosion cracking).

Material Test Matrix

<u>Material</u>	<u>Carbon</u>	<u>Ferrite</u>
CF3	.035 max.	4 - 6 FN
CF3	.035 max.	8 - 10 FN
CF3 or CF3A	.035 max.	12-14 FN
CF3 or CF3A	.035 max.	18-20 FN
CF8	.05-.06	4 - 6 FN
CF8	.05-.06	8 - 10 FN
CF8 or 8A	.05-.06	12 - 14 FN
CF8 or 8A	.05-.06	18 - 20 FN
CF8	.07-.08	4 - 6 FN
CF8	.07-.08	8 - 10 FN
CF8 or 8A	.07-.08	12 - 14 FN
CF8 or 8A	.07-.08	18 - 20 FN

Heat Treatment 1150°F - 24 Hours

90014012

In conclusion, General Electric finds it inconsistent to class all cast austenitic stainless steel with  $\geq 5\%$  ferrite (except CF3 castings) along with Type 304 stainless steel. This overly restrictive classification may result in vastly increased inspection requirements for castings - large amounts of unwarranted radiation exposure to NDT inspectors will result. General Electric, therefore, strongly recommends that all cast austenitic stainless steels with  $\geq 5\%$  ferrite be included in the list of "corrosion resistant materials."

90014013

## APPENDIX C

### Nitrogen in Austenitic Stainless Steels

G.E. strongly urges acceptance of a nitrogen content of .16% by the NRC. A .10% nitrogen content is currently accepted as a maximum impurity level for this element in most normal grades of stainless steels.

Several conferences have been held by the ASTM on the effect of trace elements including nitrogen on the properties of stainless steels (1,2,3). Kovoch (3) has shown that for heats of equivalent carbon levels, increasing the nitrogen content up to about .25% retards sensitization and reduces the crack growth rate in boiling  $MgCl_2$ . Rabbe and Heritier (3) of Cruesot-Loire have verified that increased nitrogen contents, optimally about .16% improve IGSCC resistance in austenitic steels. Columbier and Hochman have shown reduced crack depth in the Strauss test with increasing Nitrogen content up to .16%.

Steels where low carbon grades are used to achieve necessary stress corrosion resistance generally have lower mechanical properties. Nitrogen improves mechanical properties and SCC resistance as well.

In recognition of the above, "LN" grades of 304 and 316, high nitrogen (.16%) and low carbon, have been accepted by ASTM and specifications for these materials are in progress.

In view of the desirable mechanical and SCC resistant properties of these materials, change of NUREG 0313 to allow a slightly higher nitrogen content (.16%) is justified.

### REFERENCES

- 1) ASTM STP 418, 1967, Effects of Residual Elements on Properties of Austenitic Stainless Steels.
- 2) ASTM STP, 1973, Elevated Temperature Properties as influenced by Nitrogen Additions to Types 304 and 316 Austenitic Stainless Steels.
- 3) ASTM STP 679, Nov. 1977, Properties of Austenitic Stainless Steels and Their Weld Metals Influence of Slightly Chemical Variations.
- 4) Stainless and Heat Resisting Steels 1967, Columbier and Hochman.  
(portions Attached)

90014014

heat treatment was selected as giving a criterion of absolute immunity, and with a view to distinguishing sensitivity between the effects of very small differences in carbon content [28]. The procedure is capable of detecting sensitivity to intergranular corrosion under extremely severe conditions such as are seldom encountered in practice, including the effects of permanent exposure to a corrosive medium above 400°C.

Nickel influences the limiting carbon content; as the Ni content of an austenitic 18% Cr steel is raised from 9 to 13%, the carbon content must be reduced from 0.025 to 0.016% to maintain absolute immunity (Fig. 3.15).

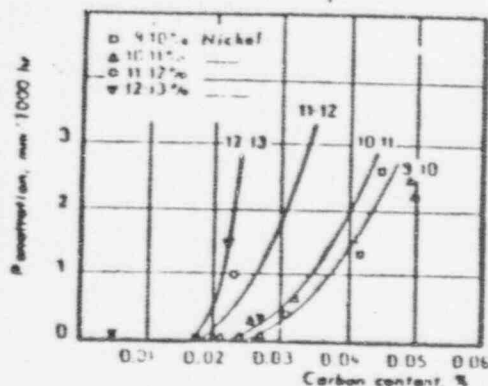


Fig. 3.15 Influence of carbon on the depth of penetration of intergranular corrosion in austenitic steel with 18% Cr, 0.01-0.05% N and the indicated nickel contents. Tested in boiling copper sulphate reagent after cooling in air from 1075°C and re-heating for 100 hours at 550°C (Binder)

If the chromium content is raised to 20-25%, the nickel content must be raised in proportion to it in order to retain the fully austenitic structure, and the beneficial effects of the increased chromium are nullified by the adverse effects of the nickel. As a result, fully austenitic steels with 25% Cr can tolerate no more carbon than those with only 18% Cr, if the criterion of immunity to intergranular corrosion is extremely severe, as in this case, i.e. absolute immunity throughout a 700-hour test in the reagent following 100 hours sensitization at 550°C.

According to the same test (100 hours at 550°C), nitrogen is harmful in the range 0.02-0.06%, with a maximum effect at about 0.04%; at nitrogen contents exceeding 0.1% however, no adverse effects can be detected (Fig. 3.16). A different picture emerges [29] when the re-heating temperature used is 650°C; the steel becomes increasingly sensitive as the nitrogen content is raised from 0.05 to 0.25% (Fig. 3.17), but the effect is extremely slight at chromium contents approaching 20%.

The fact that nitrogen can be added in appreciable amounts to austenitic steels with 20% Cr without causing any harmful effects can have important practical applications. Nitrogen is a powerful austenite stabilizer and can replace part of the nickel in austenitic

Fig. 3.16 Influence of nitrogen on the depth of intergranular corrosion in austenitic steel with 0.02-0.3% C and the indicated nickel contents. Tests in boiling copper sulphate reagent after cooling in air from 1075°C and re-heating for 100 hours at 550°C (Binder)

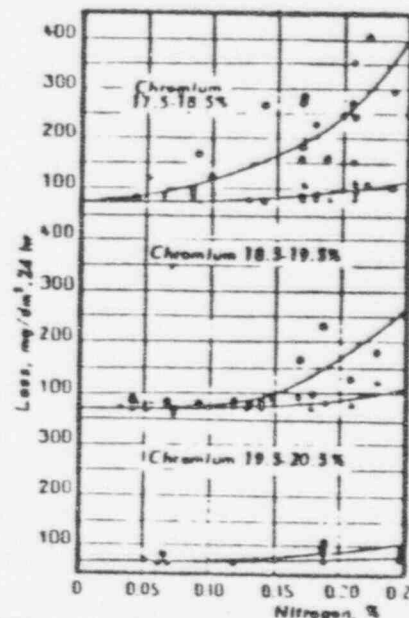
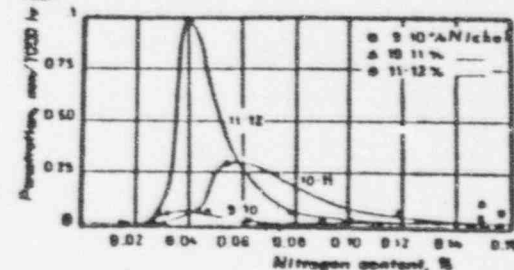


Fig. 3.17 Influence of nitrogen on the resistance of austenitic steels in boiling 85% nitric acid

steels; since nickel has an adverse influence, whereas high nitrogen contents can be tolerated without ill effect, it follows that a steel in which some nickel has been replaced by nitrogen should remain immune at higher carbon contents than the basic 18/8 steel.

Molybdenum raises the tolerance limit for carbon. Two steels were compared, containing 18% Cr, 15% Ni, 0.026% N, 0.45% Si and 1.4% Mn and differing only in carbon and molybdenum contents. The first contained 0.036% C and 3.1% Mo, the second 0.026% C and no molybdenum. After 100 hours sensitization at 550°C, the first steel was immune to intergranular corrosion, whereas the second was badly attacked. This is a most important fact, since it is precisely the Mo-bearing austenitic steels that are most frequently used in the media that give rise to intergranular corrosion (e.g. dilute sulphuric acid solutions).

The time of exposure to temperatures in the critical range is

APPENDIX DHeat Sink Welding (HSW)

General Electric considers HSW to be fully qualified for field application as a satisfactory means of achieving stress corrosion resistance for Type 304 stainless steels. Heat Sink Welds in General Electric's pipe testing programs have performed very successfully. It is clear from these results of General Electric and other investigations, that HSW's derive their benefit by both controlling the degree of sensitization in the HAZ and producing compressive residual stresses on the pipe ID, surface in the HAZ. Based on these results, General Electric recommends that NRC endorse the HSW technique for low stressed welds in plants and currently under construction and for repair of larger (>10 inch diameter) pipes in flowing water system of plants being operated.

Basis - Laboratory Data

Heat Sink Welding has been the subject of an intensive joint effort by General Electric, the Electric Power Research Institute (EPRI), the Bechtel Corporation, the Battelle Memorial Institute, and individual consultants and utilities over the past five years. The final report on the G.E. effort is listed as Reference 1. This report documents the following essential information:

1. Heat Sink Welding is a practical means of improving the IGSCC resistance of Type 304 stainless steel. The HSW process can be accomplished with either the spray cooling or flowing water cooling process as well as with stagnant water for vertical piping runs.
2. The benefits of Heat Sink Welding are due to the combined effect of reduced degree of material sensitization and to the favorable residual stress pattern established on the inner diameter surface. A representative illustration of the residual stresses established near piping welds for reference and HSW conditions is shown in Figure 3.2

90014016

3. The HSW process has been rigorously qualified for use in BWR's by means of extensive pipe tests in the GE pipe test laboratory. The results of these tests on HSW's with a machined surface have demonstrated to date a statistical factor of improvement of  $>15.1$  over the reference welded condition. These tests were conducted on four-inch diameter pipe test specimens (Figure 4) which represent the worst case of residual stress and sensitization anticipated to exist in the field. It is extremely significant that all of the failures of HSW's in general Electric's statistical program have occurred by a partially or wholly transgranular mode. This cracking mode has been associated previously only with low carbon stainless steel and is attributed to the severe nature of the pipe test. In effect, the HSW process has made a very susceptible high carbon heat of material behave as a low carbon heat. Note that two of these HSW pipes were tested at a very high primary axial tensile load of  $136\% \sigma_y$  and the other two were tested at  $110\% \sigma_y$ . Both of these stresses far exceed any value experienced in BWR operation.

At the lower load of  $110\% \sigma_y$  the only failure was an entirely transgranular failure unrelated to the weld HAZ.

Basis - Inspection vs. Remedy

G.E. considers that it is always preferable to improve materials and material conditions. This philosophy is based on the firm belief that even the best inspection techniques and sampling programs do not provide the highest degree of technical assurance that qualified IGSCC - resistance measures provide.

Wider implementation and, therefore, better overall stress corrosion improvement, would surely result from NRC endorsement of HSW at the earliest possible time. It is suggested that the NRC endorse HSW techniques for use now as IGSCC resistant measures.

90014017

REFERENCES

1. "Evaluation of Near Term BWR Piping Remedies" Final Report, by A. Giannuzzi & N.R. Hughes, May 1979 (Draft), Attachment B.
2. "Evaluation of Near Term BWR Piping Remedies" Final Report, by N.R. Hughes, December 1979, (Draft) summary sheet attached.

90014018



## 1. SUMMARY

Full size environmental pipe tests have been performed to evaluate the resistance to intergranular stress corrosion cracking (IGSCC) of Type-304 stainless steel piping fabricated by various alternate techniques. These techniques have been termed "near term BWR piping remedies" since they utilize existing Type-304 stainless steel piping. This program constitutes an extension of a broader program to identify and investigate piping remedies, as reported in EPRI NP-1222, May, 1979. Each of four near-term remedies have shown significant improvement in IGSCC resistance over reference Type-304 stainless steel. The four remedies are:

- |   |           |
|---|-----------|
| 1. Solution Heat Treatment After Welding              | SHT       |
| 2. Solution Heat Treated Corrosion Resistant Cladding | Shop CRC  |
| 3. As-Deposited Corrosion Resistant Cladding          | Field CRC |
| 4. Heat Sink Welding                                  | HSW       |

The remedies SHT, Field CRC and Shop CRC were tested with triplicate heats, while HSW was tested with one heat known to be susceptible to IGSCC in the reference condition. The significant finding for each of these remedies is discussed below.

### 1.1 REFERENCE CONDITION

In the reference condition, [normal heat input welding practice with inside diameter (i.d.) surface grinding after welding] all three heats were susceptible to IGSCC in pipe tests. These tests were all nominally 288°C (550°F)  $6 \pm 2$  ppm oxygen cyclic stress tests to a maximum stress of 136% of the 288°C (550°F) yield strength of each heat. Times of first failure varied significantly with carbon content and absolute stress. The most susceptible heat M7616 (0.060% carbon) resulted in a first failure at 104 hours, while the least susceptible heat 454970 (0.042% carbon) cycled without failure until 3253 hours. Failure analysis of each heat showed the cracking to be IGSCC except for a transition to transgranular mode at midwall of the heat 454970 failures. It has been concluded that this type of transgranular cracking is an artifact of the severe test and is not related to any boiling water reactor (BWR) service phenomenon.

### 1.2 SOLUTION HEAT TREATMENT

Testing of the three SHT pipes in this program has been uneventful. The three pipes accumulated 9831, 9989 and 10646 hours, without incident. Resulting factors of improvement are 89.5, 8.7 and 3.0 for heats M7616, M0063 and 454970, respectively. These factors were limited by test program length and vary significantly due to the wide differences in failure times of the reference Type-304 pipes.

### 1.3 SHOP CORROSION RESISTANT CLADDING

The performance of the Shop CRC pipes was as successful as the SHT pipes. Times on test of 9758, 8913 and 6806 hours translated to factors of improvements of 89.2, 7.7 and 1.9 for heats M7616, M0063 and 454970.

### 1.4 FIELD CORROSION RESISTANT CLADDING

Field CRC is designed to be applied in the field and therefore a small sensitized zone remains at the end of the CRC. This characteristic of CRC caused the M7616 heat to develop a throughwall crack at 714 hours, a factor of improvement of 6.5. The fact that heat M7616 is partially sensitized in the as-received condition may have contributed to this failure. The second heat in the matrix M0063 ran successfully the length of the program for a factor of improvement of 7.5 (8601 hours). The heat 454970 failed after reaching a factor of 1.9. This failure was transgranular and can be attributed to the severe cyclic nature of the pipe test. The crack which initiated in an area unrelated to the CRC, but which was cold worked by a machining operation is not considered to represent any known field loading situation.

These results with field CRC indicate that it is a remedy that can provide a significant margin improvement over Type-304 reference welds although the expected improvement is not as large as Shop CRC or SHT. Consideration of Field CRC is warranted in field fabrication situations utilizing Type-304 piping and can be expected to result in a 40-year plant life when applied to large diameter piping. The benefit of Field CRC is increased when it is shown that the material to which it is applied is not initially sensitized.

### 1.5 HEAT SINK WELDING

Rather than using three heats in the HSW matrix, two stresses and two surface conditions were utilized. These variations did provide a range of results discussed in Subsection 3.6.5. Heat sink welding showed a very favorable factor of improvement adequate to recommend it for field use. The series of welds fabricated as the current field specification recommends (no i.d. surface grinding) and tested at 136% of  $\sigma_y$  produced a first failure at 1650 hours for a factor of improvement of 15.1. Conservative calculations predict that a factor of only 4.0 is required to achieve a 40 year life in large diameter piping.

Failure analysis has provided additional insight into the benefits of HSW's. Sensitization due to welding can be greatly reduced to the point where an IGSCC susceptible heat like M7616 behaves like a low carbon heat, i.e., failure is by a partially transgranular mode. Further, when tested at 110%  $\sigma_y$ , the counter bore notch is favored over the weld heat affected zone (HAZ) as a crack location because compressive residual stress and only moderate sensitization make the HAZ an unlikely location for IGSCC initiation. The failure in the 110%  $\sigma_y$  pipe was entirely transgranular. In a test less severe than the normal Pipe Test Laboratory (PTL) cycle the achievable factor of improvement for HSW's may be greater than the 15.1 value achieved in this program.

90014020

APPENDIX ECorrosion Resistant Cladding

General Electric agrees with the apparent intent of the NRC to give credit to non-solution heat treated CRC. This discussion found in Section IIC states that field CRC may be used for repairs and for plants already under construction. This endorsement, however, is clouded by the next paragraph which states the following: "The joint design of all welds must be such that any high stress areas in the unstabilized wrought austenitic stainless steel, which may become sensitized as a result of a welding process, is not exposed to the reactor water.

Since it is recognized that field CRC may result in a slight degree of sensitization in the region at the edge of the cladding (a welding process) this requirement could be interpreted as a prohibition of field CRC even though it was just endorsed. This contradiction may be remedied by changing the phrase in the above quoted passage from "as a result of a welding process" to "as a result of butt welding."

The latest data available on field CRC which has been submitted to the NRC indicates a minimum factor of improvement of 7 in severe pipe tests. General Electric believes that these results are more adequate to support a clear NRC endorsement of the technique.

REFERENCES:

1. "Evaluation of Near Term BWR Piping Remedies" Final Report by A.Giannuzzi & N.R. Hughes, May, 1979, Attachment B.
2. "Evaluation of Near Term BWR Piping Remedies" Final Report by N.R. Hughes, December 1979, (Draft) Summary sheet attached (pages D-4, D-5).

90014021

APPENDIX FThe Electro Potentiokinetic Reactivation Test (EPR)

This test is used and accepted as superior to others tests for sensitization by the following organization:

General Electric Co.

Nuclear Energy Division

Manufacturing Division

Corporate Research and Development Laboratory

Steel Producers

Crucible

ARMCO

Universal

Nippon

Sumitomo

National Laboratories

Brookhaven

Argonne

Battelle

Technical Research Centre of Finland

Japan Atomic Energy Research Institute

Universities

U. of Dayton

U. of Delaware

U. of Florida

U. of Tokyo

Private Industry

Rockwell International Science Center

Texas Instruments

IHI (Japan)

Hitachi

Toshiba

Industrial Research Institute (Tokoyo)

Creusot - Loire

90014022

U. S. NUCLEAR REGULATORY COMMISSION  
SEVENTH WATER REACTOR SAFETY RESEARCH INFORMATION MEETING  
November 5 - 9, 1979

THE EPR METHOD FOR THE DETECTION  
OF SENSITIZATION IN STAINLESS STEELS

W. L. CLARKE  
GENERAL ELECTRIC COMPANY  
VALLECITOS NUCLEAR CENTER  
PLEASANTON, CALIFORNIA

90014023

## THE EPR METHOD FOR THE DETECTION OF SENSITIZATION IN STAINLESS STEELS

The Reactor Primary Coolant System Pipe Rupture Study was conducted for the Metallurgy and Materials Research Branch of the Division of Reactor Safety Research, USNRC. The over-all objective of the program was to improve the reliability of reactor system piping by increasing knowledge of failure causing mechanisms and by enhancing the capability for design evaluation and analysis. Toward the attainment of that objective, a program was completed to develop a quantitative method (EPR) for nondestructively measuring the degree of sensitization in Types 304, 304L, 316, and 316L stainless steels. The technique was extended to characterize weld heat affected zones and to correlate the degree of sensitization with intergranular stress corrosion cracking (IGSCC) resistance. Studies were directed toward establishing procedures for, and qualifying, a technique to obtain EPR measurements in-situ on reactor components in the field.

Initially, a study was completed to assess the feasibility of measuring degree of sensitization in Type 304 stainless steel using the EPR (Electrochemical Potentiokinetic Reactivation) technique. On the basis of that study, the EPR was determined to be a viable method for quantitatively measuring sensitization in Type 304, and was considered superior to the presently accepted chemical methods. The EPR method clearly distinguished between annealed Type 304 stainless steel, and material subjected to sensitizing heat treatments. In addition, the method was found extremely useful in characterizing weld heat affected zones, where it appears that a correlation exists between the degree of sensitization, measured by EPR, and IGSCC resistance in a BWR coolant environment.

The susceptibility to sensitization was evaluated electrochemically by developing potentiokinetic curves of a polarized sample obtained by controlled potential sweep from the passive to the active region (reactivation) in a specific electrolyte. The criteria used to distinguish between sensitized and annealed steels is the value of the activation charge,  $P_a$  (C/cm<sup>2</sup>), which is given by the integrated area below the reactivation peak of the curve. Sensitized steels are easily activated and show higher  $P_a$  values than annealed steels which are not susceptible to IGSCC. The value of the  $P_a$  measured is dependent on polarization rate, composition and temperature of the electrolyte, composition of the steel tested, sample surface

90014024

condition, and other factors. These test parameters must therefore be kept constant to study the reactivation of passive steels.

In addition to the sensitization measurements obtained for numerous heats of Types 304 and 304L stainless steel, a number of parametric studies were completed to obtain the greatest sensitivity possible. This sensitivity is necessary, especially when evaluating marginally susceptible (IGSCC) materials, such as low and medium heat input welds. Also, one of these parameters, the reactivation scan rate, was modified to permit utilization of the EPR technique for Types 316 and 316L stainless steels. The technique is currently not adoptable for the stabilized stainless steels (Types 321 and 347), but can be modified for use on certain nickel alloys. The procedures for performing an EPR test are presented as an appendix at the end of this manuscript.

The primary emphasis of more recent EPR development studies was to establish a method for nondestructive measurement of sensitization in the field. To accomplish this objective, it was necessary to design and construct a miniaturized field cell, and a portable polarization system. A number of field cells were constructed, and one design qualified for in-situ full usage. The portable polarization system was designed in conjunction with an outside vendor\*, and is currently being marketed commercially (together with the field cells). The field measurement procedures have been prepared and are being published in the final NRC program report (NUREG/CR-1095, Nov. 1979). The procedures require the evaluation of the outside of piping, consequently, judgements must be made relative to expected behavior on the inside (exposed to the coolant environment). The EPR field technique is very useful in assessing the metallurgical condition of the component base metal, and can easily identify sensitized material as-received from the supplier, or that produced during fabrication or installation. It is also possible to identify high heat input welds by measuring the weld heat affected zones on the outside of a pipe or component. However, until the EPR is used extensively so that a bulk of data is obtained for statistical analysis, no claims are currently being made relative to predictions of IGSCC resistance of weldments based solely on outside measurements.

The EPR technique is currently being used extensively as a quality control acceptance test by General Electric, Wilmington, manufacturing for all incoming Type 304, 304L, 316, and 316L materials. It is also specified for fabricated parts (forgings, flanges, etc.) where lack of sensitization in the finished product is specified. In

---

\*Instru Spec Associates  
Walnut Creek, CA



addition, a number of steel suppliers have purchased EPR equipment and are using the technique in their Quality Control for material supplied to General Electric Company for nuclear applications. It is expected that the General Electric Company services group will provide EPR field measurement services on customer request in the near future. We are currently awaiting the supply of the portable polarization equipment, and the training of field service personnel.

90014026

PREVIOUS REPORTS

## Quarterly Reports

GEAP-10207-33	January-June	1975
GEAP-10207-34	July-December	1975
GEAP-10207-35	January-March	1976
GEAP-10207-36	April-June	1976
GEAP-10207-37	July-September	1976
GEAP-10207-38	October-December	1976
GEAP-10207-39	January-March	1977
GEAP-10207-40	April-June	1977
GEAP-10207-41	July-September	1977
GEAP-10207-42	October-December	1977
NUREG/CR-0306	January-March	1978
NUREG/CR-0567	April-June	1978
NUREG/CR-0541	July-September	1978

TOPICAL REPORTS

GEAP-21382 "Detection of Sensitization in Stainless Steel Using Electrochemical Techniques", August 1976.

GEAP-12697 "Detection of Sensitization in Stainless Steel: II. EPR Method for Nondestructive Field Tests", February 1978.

NUREG/CR-1095 "The EPR Method for the Detection of Sensitization in Stainless Steels", November 1979.

90014027

PAPERS

"Detection of Sensitization in Stainless Steel Using Electrochemical Techniques", NACE Corrosion Conference, Paper No. 180, San Francisco, CA, March 1977.

"Comparative Methods for Measuring Degree of Sensitization in Stainless Steel", ASTM STP 656, 1978.

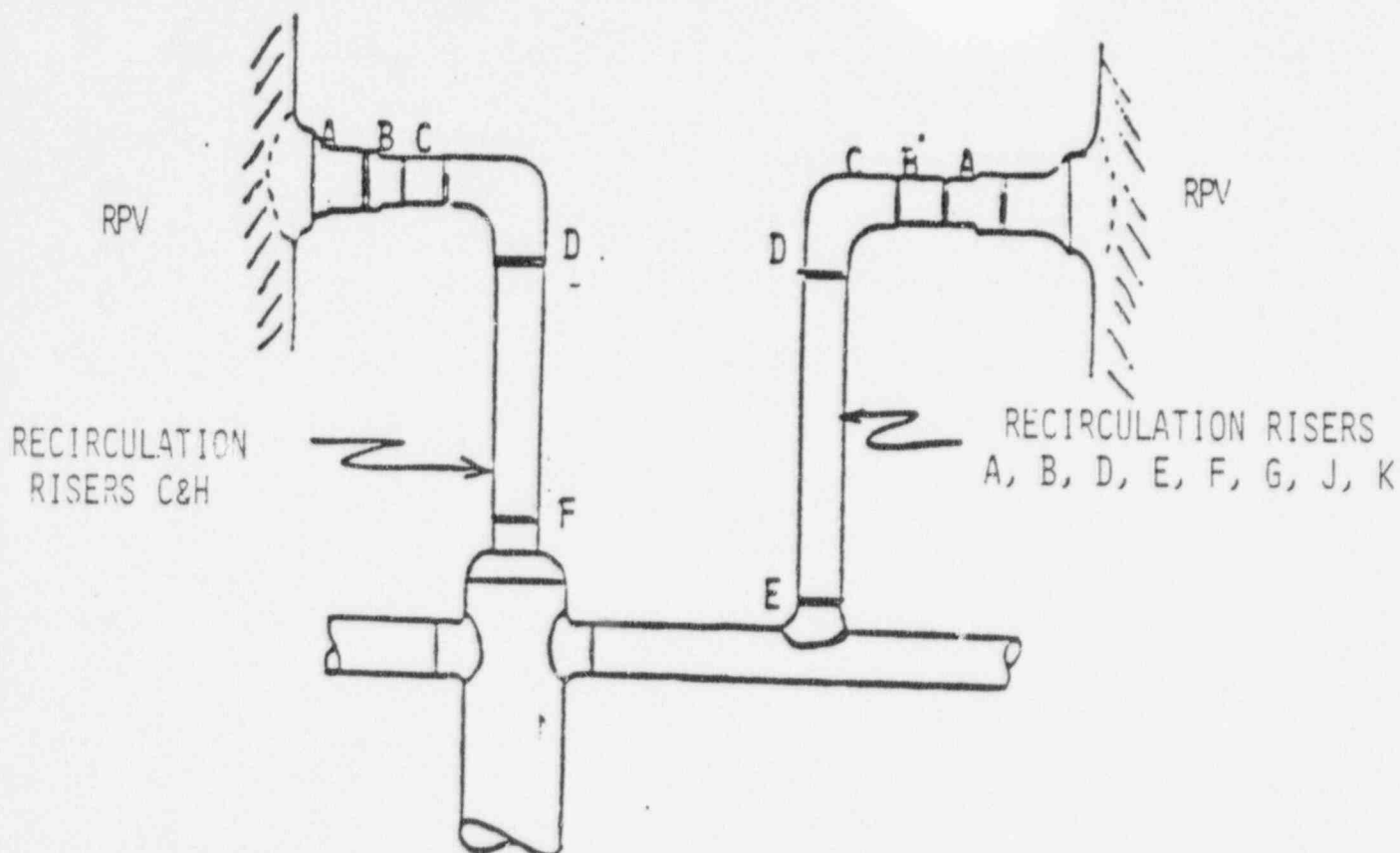
"Nondestructive Measurement of Sensitization of Stainless Steel: Relation to High Temperature Stress Corrosion Behavior", NACE Corrosion Conference, Paper No. 91, Atlanta, GA, March 1979.

"The Effects of Sensitization on the SCC Behavior of Types 316 and 316L Stainless Steel", accepted for publication, NACE Corrosion Conference, Chicago, Ill., March 1980

90014028

APPENDIX GService Sensitive Lines

Extensive inspection has already been performed on a large number of welds in the Recirculation Risers in Domestic plants. No intergranular stress corrosion cracking was detected. A list of plants inspected and results is attached.

RECIRCULATION SYSTEM RISER WELD IDENTIFICATION

SHOP MADE SEAMS  
A, C, AND D

FIELD MADE SEAMS  
B, F, AND E

WELDS WHICH HAVE EXPERIENCED IGSCC IN FOREIGN PLANTS ARE C, D, E, AND F

90014029

ASTM has accepted this test; a procedure has been written and round robin tests are in progress.

Test data obtained by G.E. (attached) shows the strong correlation between EPR results and stress corrosion testing. Other tests methods including ASTM A262 practices A and E have not shown as good a correlation with stress corrosion susceptibility.

The EPR test represents a significant advance in the technology of avoiding stress corrosion. Use of this test can predict much more accurately stress corrosion behavior in a BWR environment.<sup>(1,2)</sup> Acceptance of this test would not signal a relaxation of NRC requirements of this test which is no less severe than A262, practices A or E, only more accurate.

Use of EPR is to the advantage of all concerned; not only G.E., but also the NRC, utility owners and customers, and the general public.

#### REFERENCES:

1. "Comparative Methods for Measuring Degree of Sensitization in Stainless Steel" by Clarke, Cowan, Walker from ASTM STP 656. Attachment A.
2. "The EPR Method for the Detection of Sensitization in Stainless Steels" by W.L. Clarke, November 1979, (Attached).

90014030

RECIRCULATION RISER U.T. SURVEY  
DOMESTIC PLANTS

PLANT	NO. EXAMINED	NO. (C) (D) (E) OR (F)	YEAR STARTUP	NO. STARTUPS & SHUTDOWNS	YEAR LAST ISI
MONTICELLO	29	16	9/70	91	1976
MILLSTONE	6	6	1/71	134	1976
DRESDEN 2	20*	0	10/70	97	1976
DRESDEN 3	5	2	7/71	79	1976
QUAD CITIES 1	13	3	4/72	112	1976
QUAD CITIES 2	10	4	5/72	102	1975
PILGRIM	2	0	7/72	69	1975
COOPER	4	4	7/74	55	1976
FITZPATRICK	21	12	2/76	50	1977
DUANE ARNOLD	3	0	2/75	57	1976
BROWNS FERRY 1**	6	2	10/73	68	1976
BROWNS FERRY 2**	4	2	8/75	39	1976

- \* REPORTED EXAMINATION ON ALL (A) AND (B) WELDS 1975, 1976.
- \*\* COMMERCIAL OPERATION INTERRUPTED FOR OVER 8 MONTHS, CABLE FIRE.
- UT PROCEDURES USED AT THESE SITES WERE REVIEWED AND FOUND TO BE ADEQUATE.
- NO IGSCC DETECTED

90014031

APPENDIX HASME Code and Inspection Requirements

Recent NRC rulings in the Federal Register indicate that ASME Code application to nuclear plants has now been clarified. The NRC ruling stated that plants with Docket dates prior to July 1, 1978 would be subject to ASME Code revisions no more recent than the Summer 1975 addenda. The summer 1975 addenda to the ASME Code does not include requirements for determining such values as  $2.4 S_m$  combined primary and secondary stresses. These requirements have been added in more recent addenda. Determining these factors would constitute extreme hardship for many plants. These values are called for in NUREG 0313, Rev. 1 (for comments), page 15. Without clarification as to which plants these requirements are applicable, the NRC is imposing code requirements more recent than 1975 on plants whose Docket date is prior to July 1, 1978. This appears to be in direct contradiction with the NRC's October 9, 1979 ruling. From this ruling, which was issued concurrently with NUREG 0313, it would appear that the NRC wishes to exempt older plants from meeting recent code requirements, including the provisions in page 15 of NUREG 0313.

By designating certain lines "Service Sensitive", the NRC has already effectively applied In-Service Inspection Requirements to susceptible lines.

The apparent contradiction NUREG 0313 and the NRC's ruling may be readily resolved by incorporating the NRC's recent ruling in this NUREG.

90014032



factor and multiplying the factor by the difference between the quantity requested and the amount approved for the farm in paragraph (e)(5)(ii)(B) of this section.

(iii) Price support will be available on baled tobacco at auction sales during the same period that price support is offered on burley tobacco tied in hands in the traditional manner.

(iv) Identification cards for tobacco approved for marketing in bales with price support. A Baled Burley Tobacco Identification card showing 110 percent of the pounds of baled tobacco approved for marketing with price support shall be issued for each farm for which approval is given. The identification card together with the 1979 burley tobacco marketing card shall be used to identify any baled tobacco for which price support is desired. Separate sale bills marked "Baled Burley" shall be prepared by the warehouse to identify sales of baled burley tobacco. Each bale in the lot shall be properly identified by a card, tag, or other identification attached thereto, showing the basket number. In addition the warehouse shall mark "No Price Support" on the basket ticket and on a sale bill for any baled tobacco not identified by an identification card. A separate basket ticket and sale bill marked "No Price Support" shall be prepared for that quantity of baled tobacco weighed in that is in excess of the balance of the pounds shown on the identification card.

(v) Specification of bales:

(A) Bales accepted for price support must be approximately 1x2x3 feet in size.

(B) The leaves in bales accepted for price support must be unbed and oriented.

(vi) Grade loan rates for tobacco delivered for price support. The grade loan rates for baled burley tobacco will be the same as the grade loan rates to be established for 1979-crop burley tobacco tied in hands in the traditional manner.

#### 1464.7 Eligible producers.

(a) \*\*\*

(5) The producer has complied with any certification he/she may have executed with respect to any baled 1979-crop burley tobacco delivered for price support.

Note: This final rule has been reviewed under the USDA criteria established to implement Executive Order 12044, "Improving Government Relations." A determination has been made that this action should not be classified "significant" under

those criteria. A Final Impact Statement has been prepared and is available from Robert L. Tarnay, Price Support and Loan Division, Room 3734-South Building, P.O. Box 2415, Washington, D.C. 20250.

Signed at Washington, D.C., on September 28, 1979.

Weldon R. Denny,

Acting Executive Vice President, Commodity Credit Corporation.

OFFICE OF THE VICE PRESIDENT, COMMODITY CREDIT CORPORATION

BELLING CODE 2415-25-25

## NUCLEAR REGULATORY COMMISSION

### 10 CFR Part 50

Domestic Licensing of Production and Utilization Facilities; Codes and Standards for Nuclear Powerplants

AGENCY: U.S. Nuclear Regulatory Commission.

ACTION: Final rule.

**SUMMARY:** The Nuclear Regulatory Commission is amending its regulation, "Codes and Standards," to incorporate by reference a new edition and addenda of a national code that provides rules for the construction of nuclear powerplant components. This amendment provides for the use of updated methods in nuclear powerplant construction.

**EFFECTIVE DATE:** November 1, 1979.

**FOR FURTHER INFORMATION CONTACT:** Mr. A. Teboada, Office of Standards Development, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555, (301-443-5995).

**SUPPLEMENTARY INFORMATION:** On December 19, 1978, the Nuclear Regulatory Commission published in the Federal Register (43 FR 56825) a proposed amendment to its regulations, 10 CFR Part 50, "Domestic Licensing of Production and Utilization Facilities," to incorporate by reference new addenda to the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code. The proposed amendment to 10 CFR 50.55a would incorporate by reference the Winter 1977 addenda and the Summer 1978 addenda to Section III of the ASME Boiler and Pressure Vessel Code and also contains minor and editorial changes.

The 1977 Edition of Section XI, "Rules for Inservice Inspection of Nuclear Power Plant Components," of the ASME Code and Section XI addenda since the Summer 1975 Addenda have been evaluated by the staff and are being referenced with modifications in a separate amendment to the regulations.

The proposed amendments also included minor and editorial changes to

10 CFR 50.55a to make references to Section III in the regulations consistent with changes to Section III in the Winter 1977 Addenda. These changes in Section III of the ASME Code relate to the method for determining the edition and addenda applicable to components of the reactor coolant pressure boundary. The code presently provides that components meet the requirements of editions or addenda in effect on the date of purchase order of the components. Since the issuance of the Winter 1977 addenda, the code rules for selecting the applicable edition and addenda are more flexible. Under these rules, the licensee may establish the date of the code edition and addenda to be applied to a component. These dates may be the same for all components of a nuclear powerplant to accommodate standardization, but in no case may the dates be earlier than three years prior to the docket date for the application for the nuclear powerplant construction permit. These rules also permit more current code editions and addenda to be used. The proposed amendment would modify § 50.55a to be consistent with these changes in the code but would retain some restrictions in the regulations on the use of editions and addenda issued prior to the Winter 1972 Addenda.

Interested persons were invited to submit written comments for consideration in connection with the proposed amendment by January 17, 1979. Four letters were received in response to the notice of proposed rulemaking. In general the letters supported the proposed amendment and did not contain substantive negative comments. However, several suggestions for changes to the proposed amendment were made. Three of the letters suggested that the second sentence of proposed paragraph (b)(1) is ambiguous and should be clarified or deleted. This sentence states that "the edition and addenda selected for Section III for a given component also establishes a requirement that the identical edition or addenda be applicable to all other sections of the ASME Code used for the construction of the components".

The Commission agrees that the sentence in question is ambiguous and does not adequately describe, as intended, the code position on applicability of other sections of the code referenced in Section III. As written, the proposed rule could be interpreted to prohibit the use of newer editions and addenda of referenced sections and to require unnecessary additional welding qualification tests;

POOR ORIGINAL

90014033

interpretations not intended. The intent of this sentence was simply to describe the code position on the subject. Since the ASME Code has recently published an amplification of their position in Volume 3 of their publication, "Interpretations" (111-1-78-50) dated 1978, the sentence in question is not needed and has been deleted in the notice of rulemaking.

In the fourth letter received, the suggestion was made that footnote 5, which describes the code provisions for implementation of Section III, be amended to allow for retroactive implementation of paragraph NCA-1140 of Section III of the ASME Code which governs code edition and addenda applicability. These provisions permit new components of nuclear power plants to be constructed to an edition or addenda, to be determined by the owner of the plant, retroactive to the Winter 1977 Addenda. Footnote 5 was not revised because the Commission considers the ASME Code provisions and footnote 5 which cover this point to be adequate and appropriate. Using these rules, the principle of standardization may be applied to new construction but limited to editions and addenda since Winter 1977.

Pursuant to the Atomic Energy Act of 1954, as amended, the Energy Reorganization Act of 1974, as amended and Sections 552 and 553 of Title 5 of the United States Code, the following amendments to Title 10 Chapter 1, Code of Federal Regulations, Part 50, are published as a document subject to codification.

1. In § 50.55a of 10 CFR Part 50, paragraphs (b)(1), (c)(3), (d)(3), (e)(3), and (f)(3) are revised to read as follows:

**50.55a Codes and standards.**

Each operating license for a boiling or pressurized water-cooled nuclear power facility shall be subject to the conditions in paragraph (g) of this section and each construction permit for a utilization facility shall be subject to the following conditions in addition to those specified in § 50.55:

(b)(1) As used in this section, references to Section III of the ASME Boiler and Pressure Vessel Code refer to Section III, Division 1, and include editions through the 1977 Edition and addenda through the Summer 1978 Addenda.

These incorporations by reference provisions are approved by the Director of the Federal Register on March 17, 1972, May 4, 1972, and January 7, 1973.

**(c) Pressure vessels:**

(3) For construction permits issued on or after July 1, 1974, pressure vessels which are part of the reactor coolant pressure boundary shall meet the requirements for Class 1 components set forth in Section III of the ASME Boiler and Pressure Vessel Code. *Provided*, That the ASME Code provisions applied to the pressure vessels shall be no earlier than those of the Summer 1972 Addenda of the 1971 edition.

**(d) Piping:**

(3) For construction permits issued on or after July 1, 1974, piping which is part of the reactor coolant pressure boundary shall meet the requirements for Class 1 components set forth in Section III of the ASME Boiler and Pressure Vessel Code. *Provided*, That the ASME Code provisions applied to the piping shall be no earlier than those of Winter 1972 Addenda of the 1971 edition.

**(e) Pumps:**

(3) For construction permits issued on or after July 1, 1974, pumps which are part of the reactor coolant pressure boundary shall meet the requirements for Class 1 components set forth in Section III of the ASME Boiler and Pressure Vessel Code. *Provided*, That the ASME Code provisions applied to the pumps shall be no earlier than those of the Winter 1972 Addenda of the 1971 edition.

**(f) Valves:**

(3) For construction permits issued on or after July 1, 1974, valves which are part of the reactor coolant pressure boundary shall meet the requirements set forth in Section III of the ASME Boiler and Pressure Vessel Code. *Provided*, That the ASME Code provisions applied to the valves shall be no earlier than those of the Winter 1972 Addenda of the 1971 edition.

2. Footnotes 4, 5, and 6 to § 50.55a are revised to read as follows:

\*USAS and ASME Code addenda issued prior to the Winter 1977 Addenda are considered to be "in effect" or "effective" 6 months after their date of issuance and after they are incorporated by reference in paragraph (b) of this section. Addenda to the ASME Code issued after the Summer 1977 Addenda are considered to be "in effect" or "effective" after the date of publication of the addenda and after they are incorporated by reference in paragraph (b) of this section.

\*For ASME Code Editions and Addenda issued prior to the Winter 1977 Addenda, the Code Edition and Addenda applicable to the component is governed by the order or contract date for the component, not the contract date for the nuclear energy system. For the Winter 1977 addenda and subsequent editions and addenda the method for determining the applicable Code editions and addenda is contained in Paragraph NCA 1140 of Section III of the ASME Code.

\*ASME Code cases which have been determined suitable for use by the Commission staff are listed in NRC Regulatory Guide 1.84, "Code Case Acceptability—ASME Section III Design and Fabrication" and NRC Regulatory Guide 1.83, "Code Case Acceptability—ASME Section III Materials." The use of other Code cases may be authorized by the Commission upon request pursuant to § 50.55a(e)(2)(U).

(Secs. 103, 104, 161, Pub. L. 93-703, 98 Stat. 806, 837, 948 (42 U.S.C. 2133, 2134, 2201(f))

Dated at Washington, D.C. this 17th day of September 1979.

For the Nuclear Regulatory Commission.

Lee V. Gossick,

Executive Director for Operations.

(FR Doc. 79-21118 Filed 10-4-79; 8:45 am)

BILLING CODE 7550-01-01

**10 CFR Part 50**

Domestic Licensing of Production and Utilization Facilities; Codes and Standards for Nuclear Powerplants

AGENCY: U.S. Nuclear Regulatory Commission.

ACTION: Final rule.

**SUMMARY:** The Nuclear Regulatory Commission is amending its regulation, "Codes and Standards," to incorporate by reference a new edition and addenda of a national code that provides rules for the inservice inspection of nuclear power plant components. This amendment provides for the use of updated methods in nuclear power plant inspection.

**EFFECTIVE DATE:** November 1, 1979.

**FOR FURTHER INFORMATION CONTACT:** Mr. A. Taboada, Office of Standards Development, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555, (301-443-3997).

**SUPPLEMENTARY INFORMATION:** On January 18, 1979, the Nuclear Regulatory Commission published in the Federal Register (44 FR 3719) a proposed amendment to its regulations, 10 CFR Part 50, "Domestic Licensing of Production and Utilization Facilities," to incorporate by reference new addenda to a referenced section of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code. The amendment to 10 CFR 50.55a

POOR ORIGINAL

90014034

would incorporate by reference the 1977 Edition and addenda through the Summer 1978 Addenda to Section XI, "Inservice Inspection of Nuclear Power Plant Components," of the ASME Code with modifications.

The 1977 Edition of Section XI and addenda issued from Winter 1975 Addenda through the Winter 1977 Addenda, which had not been incorporated by reference in § 50.55a, contain several major changes to the code which, if incorporated into the NRC's regulations, would significantly reduce the examination requirements of inservice inspection programs presently required by the Commission for the reactor coolant pressure boundary and for systems required for safe shutdown of the reactor. It was the determination of the Commission that this edition and these addenda would be acceptable for incorporation by reference into the regulations only with appropriate modifications to retain those requirements considered necessary for an acceptable inservice inspection program.

In this regard, the Summer 1978 Addenda provide such modifications to Section XI of the ASME Code. Examination requirements removed from the code by the Winter 1975 Addenda through the Winter 1977 Addenda, but still required by the regulations, have either been restored or been superseded by provisions considered to be improvements.

In light of the changes in the Summer 1978 Addenda, the Commission is amending § 50.55a to incorporate by reference the 1977 Edition of Section XI of the ASME Code and Addenda through the Summer 1978 Addenda. Certain limitations and modifications to Section XI of the Code are included in the amendment to address the applicability of specific editions and addenda and to provide for flexibility and consistency in the implementation of the Code. The limitations and modifications include the following:

1. The applicability of certain code addenda to Section XI of the ASME Code is qualified to assure that appropriate inservice examination requirements are included in inservice inspection programs for nuclear facilities. These requirements were removed from Section XI of the Code in the Winter 1975 Addenda and have either been restored or have been superseded by acceptable alternatives in the Summer 1978 Addenda. The amendment, in effect, requires the application of the Summer 1978 Addenda to those inservice inspection programs that apply editions and addenda of Section XI from the Winter

1975 Addenda through the Winter 1977 Addenda.

2. The amendment provides alternatives to the Code requirements for determining the extent and frequency of inservice inspection of pipe welds. Operating facilities and facilities in the construction stages with inservice inspection programs (facilities with applications for construction permits docketed prior to July 1, 1978) may apply the Summer 1975 Addenda in lieu of later Addenda for determining the examination program for Code Class 1 and Code Class 2 pipe welds. Only code addenda incorporated by reference in § 50.55a may be used by facility licensees for inspection programs, and the Summer 1975 Addenda are the last addenda so incorporated prior to this amendment. By applying this option, operating facilities with ongoing inservice inspection programs would have continuity in the extent and frequency of examinations for pipe welds. The amendment also provides for the use of the Summer 1975 Addenda for establishing the pipe welds to be examined in the Residual Heat Removal System, the Emergency Core Cooling System, and the Containment Heat Removal System pending the development of new code provisions with sampling plans for these systems.

3. Provisions added to article IWB-2000 of Section XI of the ASME Code by the Winter 1975 Addenda contained, for the first time, requirements for inservice inspection of steam generator tubing. However, it has been the practice of the Commission to include detailed provisions for the inservice inspection of steam generator tubing in the technical specifications for a specific reactor. The potential for conflicting requirements would exist if these code requirements were incorporated by reference into the regulations without appropriate modifications. Since the provisions in the technical specifications approved by the Commission are, in general, more complete and more current, the amendment requires that where differences between the code requirements in article IWB-2000 and the technical specifications for a particular reactor exist, the inservice inspection program for steam generator tubing shall be governed by the requirements in the technical specifications.

In addition to incorporation by reference the new ASME Code edition and addenda with modifications, the Commission has made several minor and clarifying amendments to § 50.55a. These include a change in the time interval for revising programs for

inservice examination of components and for testing pumps and valves to make this interval consistent with the inservice inspection interval in Section XI of the ASME Code. The interval for revising inservice inspection programs for operating plants is extended from 40 and 20 months to 120 months. Such a change makes the regulation more practical to implement and saves time and effort for both the NRC and the licensee without an increased risk to the public health and safety. Extending the period for revising the program is not considered a significant relaxation of safety requirements since Section XI is a relatively mature code and new code changes generally deal with practical considerations of implementation or the application of new developments. New code changes do not normally modify the safety aspects of the code. Further, as stated in § 50.55a, the Commission may impose new code requirements at any time if safety considerations so dictate.

Interested persons were invited to submit to the Commission written comments on the proposed amendments for its consideration. Four letters with comments were received in response. In general, the letters supported the proposed amendment. No substantive negative comments were made. However, the letters did include requests for clarification of specific provisions of the proposed amendments. The proposed provisions in question have been clarified in the amendment.

Copies of the comments along with the comment analysis supporting this amendment are available for public inspection at the Commission's Public Document Room, 1717 H Street, NW., Washington, D.C. Single copies of the comments and comment analysis may be obtained upon request from A. Taboada, Office of Standards Development, U.S. Nuclear Regulatory Commission, Washington, D.C. 20535.

Pursuant to the Atomic Energy Act of 1954, as amended, the Energy Reorganization Act of 1974, as amended, and sections 552 and 553 of title 5 of the United States Code, the following amendments to Title 10, Chapter 1, Part 50 of the Code of Federal Regulations are published as a document subject to codification.

In § 50.55a of 10 CFR Part 50, paragraph (g)(4)(v) is deleted; paragraphs (g)(2) and (g)(3)(v) are amended by deleting the words "become effective" and substituting therefor "are incorporated by reference in paragraph (b) of this section, subject to the limitations and modifications listed therein"; and paragraphs (b)(2),

POOR ORIGINAL

90014035



and (g)(4)(i) through (g)(4)(iv) are revised to read as follows:

**§ 50.55a Codes and standards.**

Each operating license for a boiling or pressurized water-cooled nuclear power facility shall be subject to the conditions in paragraph (g) of this section and each construction permit for a utilization facility shall be subject to the following conditions in addition to those specified in § 50.53:

(b)(2) As used in this section, references to Section XI of the ASME Boiler and Pressure Vessel Code refer to Section XI, Division 1 and include editions through the 1977 Edition and Addenda through the Summer 1978 Addenda, subject to the following limitations and modifications:

(i) *Applicability of specific editions and addenda.* When applying the 1974 Edition only the addenda through the Summer 1975 Addenda may be used. When applying the 1977 Edition all of the addenda through the Summer 1978 Addenda must also be used.

(ii) *Pressure-retaining welds in ASME Code Class 1 piping (applies to Table NB-2500 and FWB-2500-1 and category B-7).* If the facility's application for a construction permit is docketed prior to July 1, 1976, the extent of examination for Code Class 1 pipe welds may be determined by the requirements of Table FWB-2500 and Table NB-2500 Category B-7 of Section I of the ASME Code in the 1974 Edition and addenda through the Summer 1975 Addenda or other requirements the Commission may adopt.

(iii) *Steam generator tubing (modifies rule FWB-2000).* If the technical specifications of a nuclear power plant include surveillance requirements for steam generators different than those in rule FWB-2000, the inservice inspection program for steam generator tubing shall be governed by the requirements in the technical specifications.

(iv) *Pressure-retaining welds in ASME Code Class 2 piping (applies to Tables C-2520 or FWC-2520-1, Category C-1A) Appropriate Code Class 2 pipe welds in Residual Heat Removal Systems, Emergency Core Cooling Systems, and Containment Heat Removal Systems, shall be examined.* The extent of examination for these items shall be determined by the requirements of paragraph FWC-1220, Table FWC-1220 Category C-F and C-G, and paragraph FWC-2411 in the 1974

Edition and Addenda through the Summer 1975 Addenda of Section XI of the ASME Code.

(B) For a nuclear power plant whose application for a construction permit is docketed prior to July 1, 1976, the extent of examination for Code Class 2 pipe welds may be determined by the requirements of paragraph FWC-1220, Table FWC-2520 Category C-F and C-G and paragraph FWC-2411 in the 1974 Edition and Addenda through the Summer 1975 Addenda of Section XI of the ASME Code or other requirements the Commission may adopt.

**(g) Inservice Inspection Requirements:**

(4) Throughout the service life of a boiling or pressurized water-cooled nuclear power facility, components (including supports) which are classified as ASME Code Class 1, Class 2 and Class 3 shall meet the requirements, except design and access provisions and preservice examination requirements, set forth in Section XI of editions of the ASME Boiler and Pressure Vessel Code and Addenda that become effective subsequent to editions specified in paragraphs (g)(2) and (g)(3) of this section and are incorporated by reference in paragraph (b) of this section, to the extent practical within the limitations of design, geometry and materials of construction of the components.

(i) Inservice examinations of components, inservice tests to verify operational readiness of pumps and valves whose function is required for safety, and system pressure tests, conducted during the initial 120-month inspection interval shall comply with the requirements in the latest edition and addenda of the Code incorporated by reference in paragraph (b) of this section on the date 12 months prior to the date of issuance of the operating license, subject to the limitations and modifications listed in paragraph (b) of this section.

(ii) Inservice examinations of components, inservice tests to verify operational readiness of pumps and valves whose function is required for safety, and system pressure tests, conducted during successive 120-month inspection intervals shall comply with the requirements of the latest edition and addenda of the Code incorporated by reference in paragraph (b) of this section 12 months prior to the start of the 120-month inspection interval, subject to the limitations and modifications listed in paragraph (b) of this section.

(iii) For a facility whose operating license was issued prior to March 1, 1976, the provisions of paragraph (g)(4) of this section are effective after September 1, 1976, at the start of the next one third of a 120 month inspection interval. During that third of an inspection interval and the remainder of the inspection interval, the inservice examinations of components, tests to verify operational readiness of pumps and valves whose function is required for safety, and system pressure tests, for such facilities shall comply with the requirements in the latest edition and addenda of the Code incorporated by reference in paragraph (b) of this section on the date 12 months prior to the start of that third of an inspection interval, subject to the limitations and modifications listed in paragraph (b) of this section.

(iv) Inservice examinations of components, tests of pumps and valves, and system pressure tests, may meet the requirements set forth in subsequent editions and addenda that are incorporated by reference in paragraph (b) of this section, subject to the limitations and modifications listed in paragraph (b) of this section, and subject to Commission approval. Portions of editions or addenda may be used provided that all related requirements of the respective editions or addenda are met.

(Secs. 103, 104, 3811, Pub. L. 93-703, 66 Stat. 936, 937, 944 (42 U.S.C. 2031, 2034, 2201(7)))

Dated at Washington, D.C. this 17th day of September 1979.

For the Nuclear Regulatory Commission.

Lee V. Gosnick,

Executive Director for Operations.

(FR Doc. 79-2147 Filed 10-9-79; 8:45 am)

BILLING CODE 7550-01-02

## SMALL BUSINESS ADMINISTRATION

### 13 CFR Part 121

(Rev. 13, April 30)

Small Business Size Standards;  
Methods of Determining Small  
Business Status for Small Business  
Administration Loan Assistance

AGENCY: Small Business Administration.  
ACTION: Final rule.

**SUMMARY:** This rule defines the method for determining a firm's small business status for purposes of SBA financial assistance when a firm and its affiliated concerns are engaged in more than one industry with more than one size standard. The existing formula method has produced inequitable and

The location  
revised by U.S.  
of the Federal Register  
on 10-17-79

reference provisions were  
of the Federal Register  
on 10-17-79 and February 7, 1978.

POOR ORIGINAL

90014036

Authorized Reprint from  
Special Technical Publication 656  
Copyright  
American Society for Testing and Materials  
1916 Race Street, Philadelphia, Pa. 19103  
1978

W. L. Clarke,<sup>1</sup> R. L. Cowan,<sup>2</sup> and W. L. Walker<sup>2</sup>

## Comparative Methods for Measuring Degree of Sensitization in Stainless Steel

**REFERENCE:** Clarke, W. L., Cowan, R. L., and Walker, W. L., "Comparative Methods for Measuring Degree of Sensitization in Stainless Steel," *Intergranular Corrosion of Stainless Alloys*, ASTM STP 656, R. F. Steigerwald, Ed., American Society for Testing and Materials, 1978, pp. 99-132.

**ABSTRACT:** Three test methods for determining degree of sensitization in austenitic stainless steels have been investigated. The results clearly show that all three test methods are capable of detecting moderate-to-severe degrees of sensitization, but the electrochemical potentiokinetic reactivation technique is the most sensitive for quantitatively determining the levels of sensitization which are of primary concern for industrial use. Both the ASTM Recommended Practices for Detecting Susceptibility to Intergranular Attack in Stainless Steels (A 262-75, Practice A) and the electrochemical potentiokinetic reactivation methods appear to saturate at high degrees of sensitization, which results in a loss of discriminating power between different heats of material. While the A 262, Practice E, method does not appear to saturate and retains its discriminating power at high degrees of sensitization, it is not a suitable method for detection at the lower degrees. Of the three test methods, it appears that the electrochemical potentiokinetic reactivation test is the most suitable for determining the quantitative degree of sensitization over the levels of industrial concern.

**KEY WORDS:** stainless steels, intergranular corrosion, sensitizing, electrochemistry, strains, loads, stress corrosion cracking

The accepted ASTM Recommended Practices for Detecting Susceptibility to Intergranular Attack in Stainless Steels (A 262-75, Practices A, E) used to detect sensitization have three major deficiencies: (a) they do not readily quantify the degree of sensitization, (b) they are not rapid (with the exception of ASTM A 262, Practice A), and (c) they are destructive (with the exception of A 262, Practice A). The accepted practices were developed to screen materials that would be subjected to highly corrosive environ-

<sup>1</sup>Principal metallurgical engineer, Vallecitos Nuclear Center, General Electric Company, Pleasanton, Calif. 94566.

<sup>2</sup>Manager, Plant Component Behavior Analysis, and principal metallurgical engineer, Plant Materials and Processes, respectively, General Electric Company, San Jose, Calif. 95125.

ments which could cause severe general intergranular attack in heavily sensitized austenitic stainless steels. In recent years, experience has shown that "moderately" sensitized materials can undergo intergranular stress-corrosion cracking (IGSCC) in environments that do not cause appreciable intergranular attack in the absence of stress [1].<sup>3</sup> Thus, a more discerning test is required for these applications. Conversely, there are applications where the use of moderately sensitized material, which could not pass the current practices for determining the presence of sensitization, would perform satisfactorily in the service environment [2]. In these cases, a manufacturer would be forced to use an extra-low-carbon or stabilized grade of material, with the associated cost or strength penalties. A test that measured the degree of sensitization, in conjunction with calibration tests in the service environment, could provide a "go/no go" materials acceptance criteria for both cases. A rapid nondestructive test would also be helpful for quality control on shop- or field-constructed components which receive thermal treatments during fabrication.

In this paper, progress in establishing a quantitative test method for measuring degree of sensitization is described. Three techniques were investigated: (a) modified A 262, Practice A, (b) modified A 262, Practice E, and (c) an electrochemical reactivation method. A fourth technique, a dynamic straining stress corrosion test [3] was used as a reference method to determine the effect of moderate amounts of sensitization on IGSCC. In this referee test, dynamic straining coupled with a very severe environment (550°F high-purity water containing 8-ppm O<sub>2</sub>) can cause intergranular fracture of mildly sensitized stainless steel.

## Experimental Procedures

### Materials

There were thirteen heats of AISI Type 304 stainless steel used in the sensitization measurements program. These heats included five heats of 10.2 cm (4 in.), Schedule 80 seamless piping; one heat of 25.4 cm (10 in.), Schedule 80 and two heats of 15.2 cm (6 in.), Schedule 80 seamless pipe; one heat of 66 cm (26 in.), Schedule 80 rolled and welded pipe; one heat each of 2.5 cm (1 in.) thick forged bar, forged plate, and hot-rolled plate; and one heat of 3.1 cm (1¼-in.) bar. The chemical composition and mechanical properties for these materials are given in Tables 1 and 2, respectively.

For the bulk of the study, specimens from twelve heats were tested in the as-received (mill-annealed (MA)) condition, and, after a low-temperature sensitization (LTS) treatment at 500°C (932°F) for 24 h in air, followed

TABLE 1—Composition of steels used in sensitization program (element, percent; nd = not determined).

Heat	C	Cr	Ni	Mn	Si	Mo	Cu	S	P	N
M7616 <sup>a</sup>	0.060	18.68	10.16	1.69	0.50	0.08	0.08	0.012	0.022	0.038
812292 <sup>b</sup>	0.069	18.18	9.14	0.74	0.58	0.19	0.17	0.014	0.019	0.034
78500 <sup>c</sup>	0.043	19.06	8.88	1.64	0.55	0.04	0.42	0.012	0.016	0.073
8082228 <sup>d</sup>	0.055	18.22	8.65	1.74	0.57	0.38	0.30	0.013	0.030	nd
M7772 <sup>e</sup>	0.050	18.81	10.15	1.80	0.38	0.11	0.15	0.015	0.026	0.034
TH5656 <sup>f</sup>	0.060	18.31	9.30	1.72	0.47	0.24	0.25	0.006	0.024	0.030
454659 <sup>g</sup>	0.045	18.40	9.76	1.25	0.57	0.23	0.07	0.015	0.018	0.029
834264 <sup>h</sup>	0.060	18.30	9.12	1.58	0.62	0.30	0.09	0.028	0.030	nd
27388 <sup>i</sup>	0.044	18.86	8.76	1.77	0.60	0.11	0.49	0.010	0.019	0.072
2P6340 <sup>j</sup>	0.040	18.66	10.30	1.65	0.53	0.20	0.12	0.016	0.022	0.033
2P6424 <sup>k</sup>	0.040	18.37	9.61	1.65	0.46	0.25	0.08	0.013	0.021	0.036
159340 <sup>l</sup>	0.070	18.32	9.35	1.75	0.40	0.41	0.10	0.017	0.035	nd
X14902 <sup>m</sup>	0.065	18.68	9.20	1.75	0.48	nd	nd	0.028	0.029	nd

<sup>a</sup>4-in., Schedule 80 seamless pipe.

<sup>b</sup>1 by 4-in. forged bar.

<sup>c</sup>6-in., Schedule 80 seamless pipe.

<sup>d</sup>1-in., hot-rolled plate.

<sup>e</sup>10-in., Schedule 80 seamless pipe.

<sup>f</sup>26-in., rolled and welded pipe.

<sup>g</sup>1 by 12-in. forged plate.

<sup>h</sup>1¼ by 1-in. bar.

Metric conversion: 1 in. = 2.5 cm.

POOR ORIGINAL

<sup>3</sup>The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 2—Mechanical properties of steels used in sensitization studies (nd = not determined).

Heat	Room Temperature			289°C (550°F)				
	YS (ksi)	UTS (ksi)	E (%)	RA (%)	YS (ksi)	UTS (ksi)	E (%)	RA (%)
M7616	41.6	91.9	73.5	74.9	28.8	69.4	40.3	71.7
M12292	36.4	91.4	82.9	81.7	30.0	66.8	44.3	73.8
785K	35.2	86.0	83.3	81.7	17.8	61.5	51.4	72.5
8082228	41.9	92.9	75.5	75.0	26.3	69.6	46.9	64.4
M7722	41.1	92.4	68.3	78.1	28.5	69.7	40.2	72.1
T700S6	37.0	90.1	66.8	83.4	24.1	66.1	39.1	75.6
54559	35.4	90.4	60.6	79.3	25.9	66.7	34.2	70.7
834204	38.5	89.5	66.0	70.0	nd	nd	nd	nd
27388	39.3	90.7	77.1	77.3	22.7	64.3	49.0	75.2
P6390	38.4	87.7	75.8	78.5	24.2	65.3	42.3	72.7
P6424	39.0	89.6	74.3	81.6	26.1	66.0	42.5	75.5
59340	35.1	90.4	77.6	75.8	24.0	68.3	48.1	70.6
X14902	38.2	92.5	76.3	81.5	26.2	67.8	41.6	75.6

by furnace cooling. This treatment was used to produce light degrees of sensitization to test the sensitivity of the various methods. Specimens from two heats (M7616 and 27388) were also tested after solution annealing at 1038°C (1900°F) for 1 h and water quenching, followed by the LTS treatment. Additionally, one specimen from heat M7616 was tested in the solution-annealed condition (solution-annealed treatment just described), without a sensitization heat treatment.

In addition, two smaller studies were performed to investigate the effects of more severe sensitization treatments. In one series of tests, specimens from six piping heats were evaluated after sensitizing 40 h at 620°C (1150 °F); the other study was conducted on specimens from a single heat of bar stock (Heat X14902). These specimens were sensitized 1, 4, 20, and 40 h at 620°C (1150°F).

### Measurement of Sensitization

**ASTM A 262, Practice A**—All test specimens were evaluated for percent ditching after oxalic acid etching according to ASTM A 262, Practice A, modified according to the procedure given in Appendix 1. The modification of the ASTM practice consisted of measuring and reporting the amount of grain boundary length that showed a ditched structure. The specimens examined were those already mounted which were repolished after electrochemical potentiokinetic reactivation (EPR) testing (discussed later).

**ASTM A 262, Practice E**—Companion specimens to those used for the A 262, Practice A, and electrochemical tests were also evaluated for sensitization using two modifications of A 262, Practice E, the acidified copper-copper sulfate test. The conventional A 262, Practice E, test gives "go/no go" results, that is, either the specimen exhibits fissuring when bent, or it does not. The first modification used was to obtain semiquantitative data by accurately measuring maximum penetration depths on tension specimens which were strained 3 to 5 percent after exposure to the test solution, and then examining metallographically prepared specimens (Appendix II).

The second method consists of measuring the penetration which occurs during the test period by means of the change in effective cross-sectional area of the specimen, by comparison of the ultimate tensile strength of the tested specimen to a specimen of the same material which has not been exposed to the test solution [4]. This treatment of the data results in a true measure of penetration during the test, regardless of grain fallout (Appendix II).

**EPR Measurements**—The degree of sensitization was quantified using the recently developed EPR method. This method consists of developing potentiokinetic curves of a polarized specimen obtained by controlled potential sweep from the passive to the active region (reactivation) in a



specific electrolyte; details of the test technique have been reported [5,6], and are presented in specification form in Appendix III. The test conditions used for the EPR measurements are given in Table 3. The criteria used to distinguish between annealed and sensitized specimens include the activation charge,  $Q$ , given by the integrated area below the reactivation peak of the curve (Fig. 1). Sensitized steels are easily activated and show high  $Q$  values, compared to annealed steels which are not susceptible to intergranular corrosion. The value  $Q$  is normalized by both specimen size and grain size as described in Appendix III. The data normalized in this fashion are called  $P_a$  and represent the charge per square centimetre of grain boundary area. This treatment of the data permits normalized direct comparisons of different heats of material which exhibit different  $Q$  values solely as a result of differences in grain size. This topic has been described in detail [5].

The EPR specimens were 0.3 by 0.3-cm sections cut from the tab ends of the uniaxial tension specimens tested for IGSCC resistance. The specimens were mounted in a Maraglas compound, so that only one face of the specimen was exposed. Electrical contact was made by spot-welding a stainless steel screw to each specimen before mounting. Finally, the mounted specimens were polished before testing so that the effect of the test (grain boundary attack) could be viewed and documented metallographically.

**IGSCC Tests**—The susceptibility to IGSCC was determined by conducting dynamic strain tests [1,3] in 289°C (550°F) high-purity (approximately 1  $\mu$ S) water containing 8-ppm dissolved oxygen. This is a very severe test and does not usually represent an environment of industrial interest. However, this extreme case was used in this investigation to maximize the potential for correlation between sensitization and IGSCC testing. In the application of these tests in industrial practice, the type correlation performed in this study would have to be undertaken in test environments of industrial interest. In this test, uniaxial tension test specimens are slowly strained to failure at 0.0008 mm/min (0.032 mil/min) in the environment. Susceptible materials generally reveal shorter failure times, lower breaking stresses, and lower reduction-in-area (RA) values compared to similar tests performed in air or inert gas. In addition, the data generated can be treated to obtain a susceptibility index,  $I_{DS}$ , with the expression

$$I_{DS} = 1 - \frac{\sigma_w(1 + E_w)}{\sigma_A(1 + E_A)}$$

where

- $\sigma_w$  = maximum breaking stress in water with  $O_2$ ,
- $\sigma_A$  = maximum breaking stress in air,
- $E_w$  = elongation in water, and
- $E_A$  = elongation in air.

TABLE 3—EPR test conditions.

Electrolyte	0.5 M $H_2SO_4$ + 0.01 M KCNS
Temperature	30°C
Specimen surface finish	1 $\mu$ m (diamond paste)
Reactivation sweep rate	6 V/h (cathodic)
Passivation potential time	+ 200 mV/2 min
Deaeration	$N_2$
Polarization system	Hokuto-Denko with Princeton Applied Research Coulometer
Data normalization	$P_a (C/cm^2) = Q (C)/GBA (cm^2)^{1/2}$

<sup>a</sup>GBA = calculated grain boundary area in sample (see Appendix III).

Greater susceptibility to IGSCC is indicated as  $I_{DS}$  approaches a value of 1. Finally, the failure mode is documented by scanning electron microscopic and metallographic examination of the fractured specimens. Test specimen gage length is typically 1.9 cm (0.75 in.).

## Results

The results of the study to evaluate degree of sensitization in twelve heats of Type 304 stainless steel in the mill-annealed condition, and after sensitizing 24 h at 500°C, are given in Table 4. Specimens from seven of the

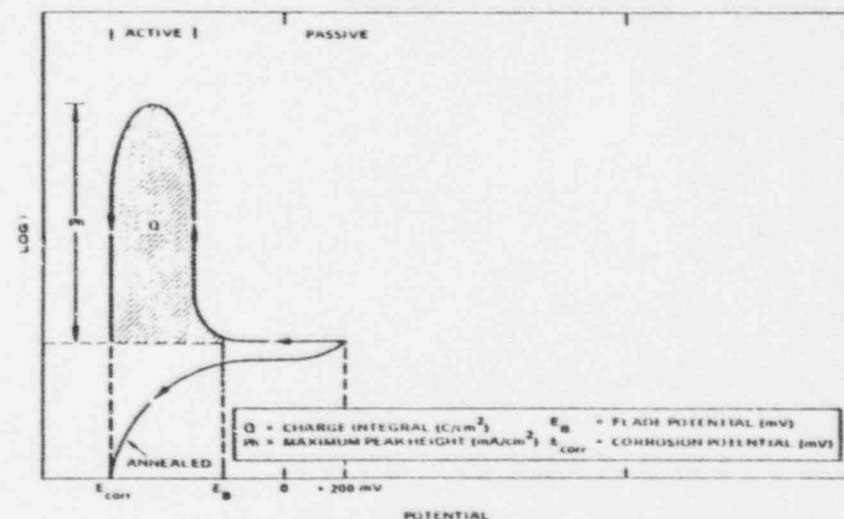


FIG. 1—Schematic of reactivation polarization curve showing parameters of interest for EPR testing.



TABLE 4—Summary of data developed to compare three methods for measuring degree of sensitization in Type 304 stainless steel with IGSCC susceptibility

Heat	Material Identification	Condition <sup>a</sup>	Degree of Sensitization <sup>a</sup>			IGSCC Susceptibility after Dynamic Strain	
			A 262-A (% Ditch)	A 262-E (mil)	EPR (C/cm <sup>2</sup> )	I <sub>DS</sub>	IGSCC <sup>c</sup>
M7616	Seamless pipe, 4-in., Schedule 80	SA	...	<0.1	0.0	0.20	no
	SA + LTS	SA	...	...	...	0.37	no
	MA	MA	40	0.2	7.3	0.69	yes
812292	Forged bar, 1-in.	MA + LTS	50	0.4 to 0.8	40.0	0.92	yes
	MA	MA	...	<0.1	2.0	0.51	no
	MA + LTS	MA	...	0.1 to 0.4	16.8	0.75	yes
76500	Seamless pipe, 6-in., Schedule 80	MA	...	<0.1	1.8	0.38	no
	MA + LTS	MA	...	0.4 to 0.8	12.0	0.65	yes
8062228	Hot rolled plate, 1-in.	MA	...	<0.1	0.4	...	...
	MA + LTS	MA	...	0.4 to 0.8	7.4	0.82	yes
M7772	Seamless pipe, 4-in., Schedule 80	MA	...	<0.1	0.0	...	...
	MA + LTS	MA	...	0.2 to 0.4	6.7	0.52	no
TH6656	Seamless pipe, 10-in., Schedule 80	MA	2	<0.1	0.0	...	...
	MA + LTS	MA	14	0.2 to 0.8	6.5	0.65	yes
454634	Seamless pipe, 4-in., Schedule 80	MA	...	<0.1	0.0	...	...
	MA + LTS	MA	...	0.8 to 1.6	7.3	0.77	yes
634204	Roll and welded pipe, 26-in., Schedule 80	MA	5	0.1	0.7	0.53	no
	MA + LTS	MA	25	0.1 to 0.2	4.6	0.71	yes
27366	Seamless pipe, 6-in., Schedule 80	SA	...	<0.1	0.0	0.36	no
	MA	MA	...	<0.1	0.4	0.59	no
	MA + LTS	MA	...	0.8 to 1.6	4.2	0.42	no
2P6396	Seamless pipe, 4-in., Schedule 80	MA	...	<0.1	0.0	...	...
	MA + LTS	MA	...	0.4 to 0.8	4.0	0.44	no
2P6424	Seamless pipe, 4-in., Schedule 80	MA	...	<0.1	0.2	...	...
	MA + LTS	MA	...	0.1 to 0.2	2.6	0.53	no
159340	Forged plate, 1-in.	MA	...	0.1	0.0	0.0	...
	MA + LTS	MA	...	0.1 to 0.2	2.7	0.55	no

<sup>a</sup>ASTM A 262, Practice E pulled 3 to 5 percent in tension after three 72-h exposures. EPR at 6 V/h and 30°C in 0.5 M H<sub>2</sub>SO<sub>4</sub> + 0.01 M KCN (GBA adjusted).

<sup>b</sup>Solution annealed (SA) = 1038°C (1900°F) / 1 h water quenched. MA = mill annealed. LTS = 500°C (932°F) / 24 h furnace cooled.

<sup>c</sup>SEM examination of fracture surfaces.

Metric conversion: 1 in. = 2.5 cm.

POOR ORIGINAL

twelve heats indicated IGSCC susceptibility after dynamic strain testing, with an  $I_{DS}$  value of 0.65 established for this heat treatment in this environment as the lower limit for susceptibility. The most extensive IGSCC noted occurred in one of the 10.2-cm (4-in.) seamless pipe heats (M7616), where IGSCC occurred even in the mill-annealed condition (Fig. 2) in this severe environment. The mill-annealed specimen did reveal some mixed mode cracking (intergranular plus transgranular), but crack initiation was always intergranular. This particular heat of material was also extremely susceptible to IGSCC in other studies [3,5] using more severe sensitization treatments. This level of susceptibility would have been predicted by degree of sensitization measurements using both A 262, Practice A, and the EPR technique. But, the EPR method provides quantitative data relative to the level of susceptibility between mill-annealed and sensitized conditions ( $P_a = 7.3$  and 40.0 C/cm<sup>2</sup> compares to  $I_{DS} = 0.69$  and 0.92, respectively), while the A 262, Practice A method shows little differentiation (40 and 50 percent ditching). The etch structures developed after the A 262, Practice A, and EPR exposures are shown in Figs. 3 and 4, respectively. As shown in Fig. 3, the extent of grain boundary ditching appears comparable for both conditions; however, the EPR-produced structures (Fig. 4) clearly delineate the difference in grain boundary attack (chromium depletion around carbide particles) [5] between the mill-annealed and lightly sensitized condition.

In contrast, the A 262, Practice E, test does not appear very sensitive at these lower degrees of sensitization. The structures developed in the mill-annealed and sensitized specimens after three 72-h exposures are shown in Fig. 5, where penetration depths after straining 3 to 5 percent can only be measured with great difficulty. Based on the data developed by A 262, Practice E, for the other eleven heats, the difference between penetration depths of 0.2 mil for the mill-annealed condition and 0.4 to 0.8 mil after sensitizing do not appear significant.

The 25.4-cm (10-in.) seamless pipe heat (TH6656) revealed the lowest level ( $I_{DS} = 0.65$ ) of IGSCC after sensitizing of the seven susceptible materials; this material was not susceptible in the mill-annealed condition. The fracture surface for this specimen after dynamic strain testing is shown in Fig. 6. Only one corner of the specimen revealed IGSCC; the remainder of the specimens failed ductilely (approximately 90 percent of the fracture surface reveals IGSCC in highly susceptible materials after severe sensitization treatments) [3]. This specimen also contained only a few secondary cracks, while highly susceptible materials, such as Heat M7616, usually develop secondary cracking too numerous for counting.

The etch structures produced in the sensitized Heat TH6656 specimen during degree of sensitization measurements using the three methods are shown in Fig. 7. Again, the A 262, Practice E, results are not sufficiently discerning to predict the IGSCC behavior. Both the A 262, Practice A, and EPR method reveal a sensitized microstructure, but only the EPR measure-



FIG. 2—Stress corrosion cracking in Type 304 stainless steel (Heat M7616) tested by dynamic strain in 289°C water with 8 ppm O<sub>2</sub> at extension rate of 0.0008 mm/min: (a) null annealed,  $\times 100$ ; (b) sensitized 24 h at 500°C, SEM,  $\times 200$ .

POOR ORIGINAL

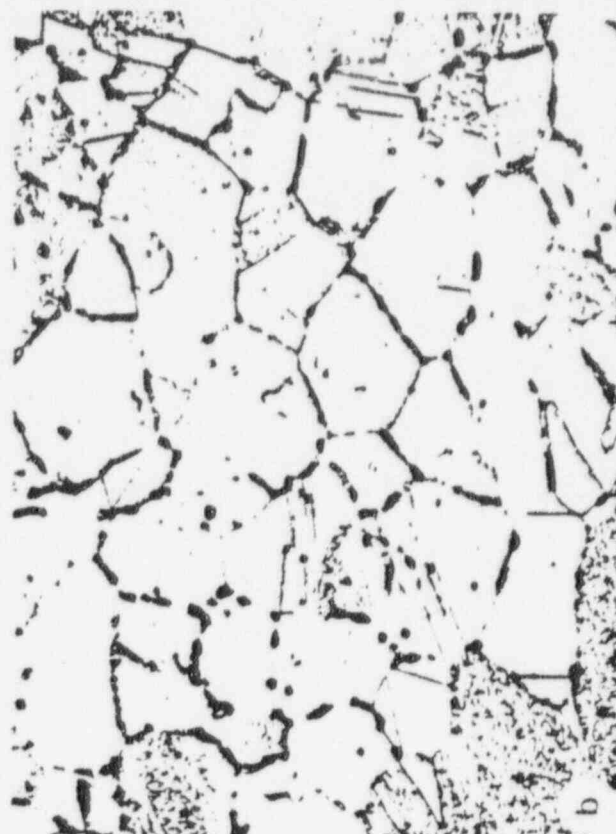


FIG. 3—ASTM A 262, Practice A etch in Type 304 stainless steel (Heat M7616): (a) null annealed (40 percent ditching),  $\times 250$ ; (b) sensitized 24 h at 500°C (30 percent ditching),  $\times 250$ .

90014042

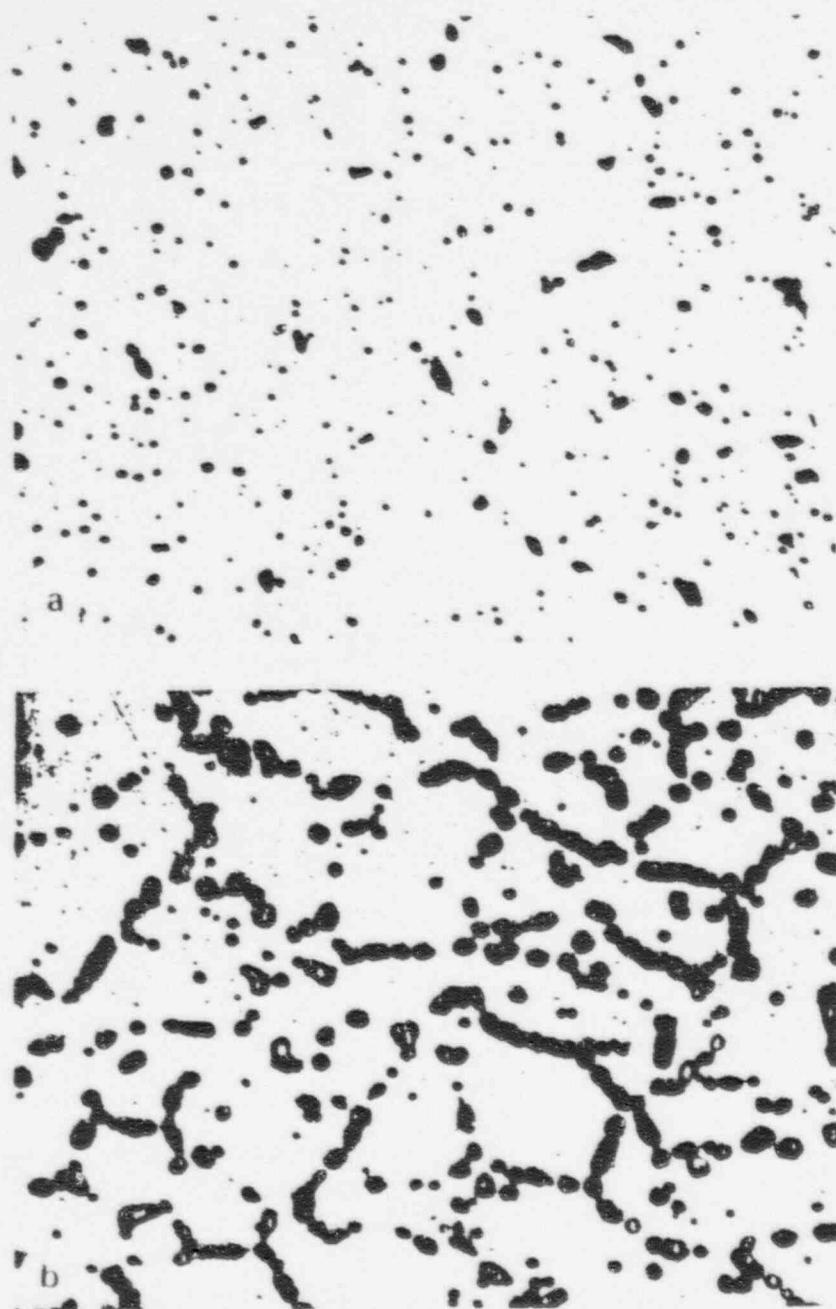


FIG. 4—Etch structure in Type 304 stainless steel (Heat M7616) after  $E_{PR}$  testing in 0.5 M  $H_2SO_4$  + 0.01 M KCNS at 30°C: (a) mill annealed ( $P_A = 7.3 C/cm^2$ ),  $\times 250$ , (b) sensitized 24 h at 500°C ( $P_A = 40 C/cm^2$ ),  $\times 200$ .

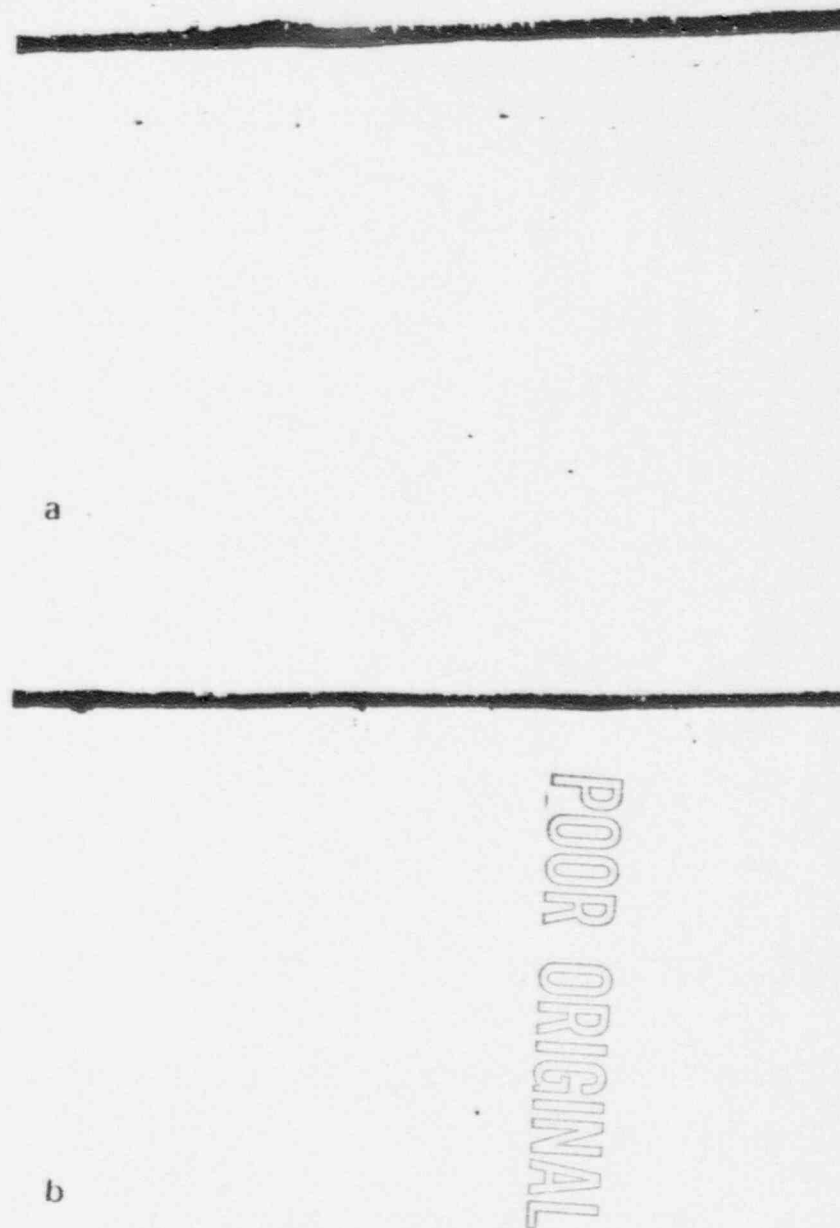


FIG. 5—ASTM A 262, Practice E attack in Type 304 stainless steel (Heat M7616) after three 72 h exposures (pulled 3 to 5 percent in tension): (a) mill annealed ( $P_A = 7.3 C/cm^2$ ),  $\times 250$ , (b) sensitized 24 h at 500°C (0.4 to 0.8 mil penetration),  $\times 250$ .

POOR ORIGINAL

90014043

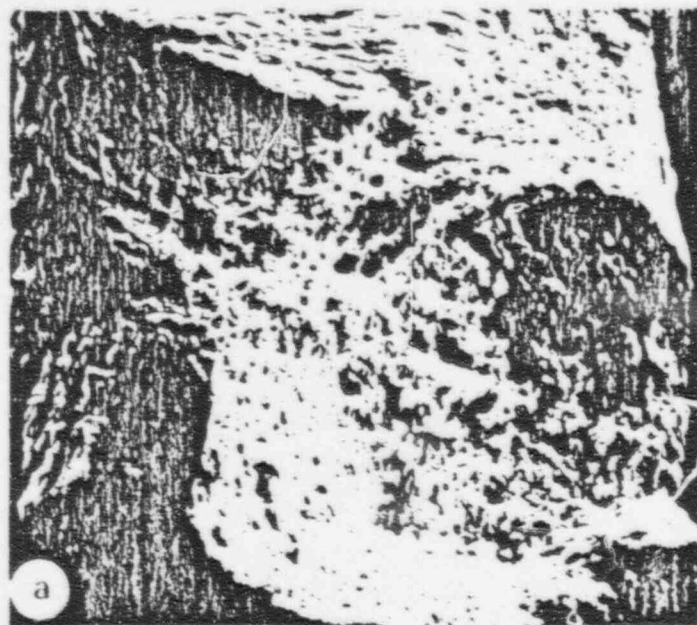


FIG. 6—Fracture surface of sensitized (500°C/24 h) Type 304 stainless steel seamless pipe (Heat TH6656) tested by dynamic strain in 289°C water with 8 ppm  $O_2$  at an extension rate of 0.0008 mm/min. (a)  $\times 40$ . (b)  $\times 300$ .

90014044

POOR ORIGINAL

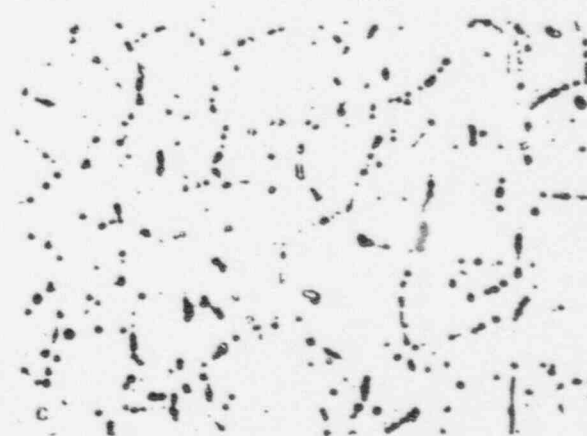
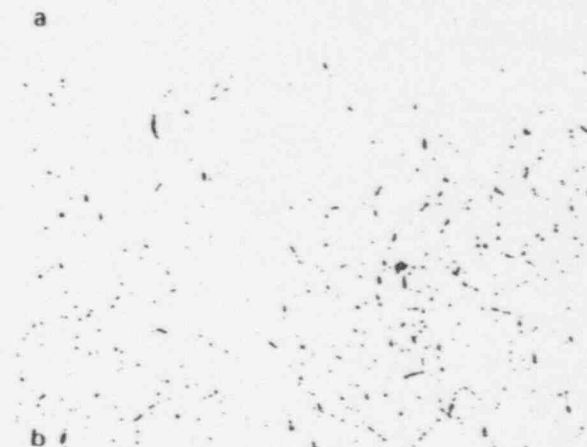


FIG. 7—Etch structures in Type 304 stainless steel seamless pipe (Heat TH6656) sensitized 24 h at 500°C and tested for degree of sensitization using three techniques. (a) A 262, Practice E (0.2 to 0.8 mil penetration),  $\times 187$ . (b) EPR ( $P_s = 6.5 \text{ C/cm}^2$ ),  $\times 89$ . (c) A 262, Practice A (14 percent ditching).



ment quantitatively ranks this heat near the lowest end of the scale of susceptible materials. According to the A 262, Practice A test, Heat TH6656 would be expected to be more susceptible to IGSCC than Heats 812292 or 78500, using only degree of sensitization as a criterion.

An intermediate level of IGSCC susceptibility occurred in a heat of 1-in. forged bar (Heat 812292) dynamically tested after the 500°C/24 h sensitization treatment. A specimen from this heat revealed IGSCC about 2 to 3 grains deep (Fig. 8) and a transgranular region below the IGSCC initiated cracks. The sensitization measurement etch structures for this heat are shown in Fig. 9. The A 262, Practice E, test indicated penetrations of 0.1 to 0.4 mil, which occurred at slip planes (Fig. 9a) produced by surface cold work during specimen machining.

The EPR test gave a relatively high value ( $P_s = 16.8 \text{ C/cm}^2$ ) for degree of sensitization, and the structure revealed a profusion of precipitated carbides (Fig. 9b). The carbides present are very small and difficult to resolve using conventional metallographic techniques. This precipitation mode probably accounts for the lack of grain boundary grooving after A 262, Practice A, testing (Fig. 9c), where the chromium carbides are presumably too small for the sensitivity of the oxalic acid test. Thus, an advantage is shown for the EPR test method, particularly for some precipitate morphologies.

Generally, the A 262 tests (Practices A and E) are not sufficiently discerning to distinguish between degrees of sensitization at these low levels of sensitization. One exception is the evaluation of the 66-cm (26-in.) rolled and welded pipe (Heat 834264), which has exhibited inconsistent behavior in other related studies [6]. Specimens from this large-grained (ASTM 3.5 compared to 5 to 5.5 for the other materials) material are highly susceptible to IGSCC but consistently yield relatively low sensitization values in the EPR test. The A 262, Practice E, test also indicates relatively low levels of sensitization, but the A 262, Practice A, test ranked this heat properly. Although the EPR technique gave a low  $P_s$  value for this heat, the post-test appearance did show considerable grooving of the grain boundaries, indicating that post-test examination of the EPR attack should be weighted in the decision process at low  $P_s$  values.

Analysis of the remainder of the data in Table 4 indicates the EPR test is more sensitive than either A 262, Practices A or E, in distinguishing and quantifying degree of sensitization at these lower levels of sensitization. It is significant that the high IGSCC susceptibility of Heat M7616 can be eliminated by solution annealing, even when the material is subsequently given the low-temperature sensitization treatment. It was difficult to solution anneal this material adequately, however, as even rapid air cooling after the 1038°C/h anneal resulted in a sensitized structure similar to the mill-annealed condition given in Table 4. Complete solution annealing was accomplished in this heat only after water quenching in the cooling chamber

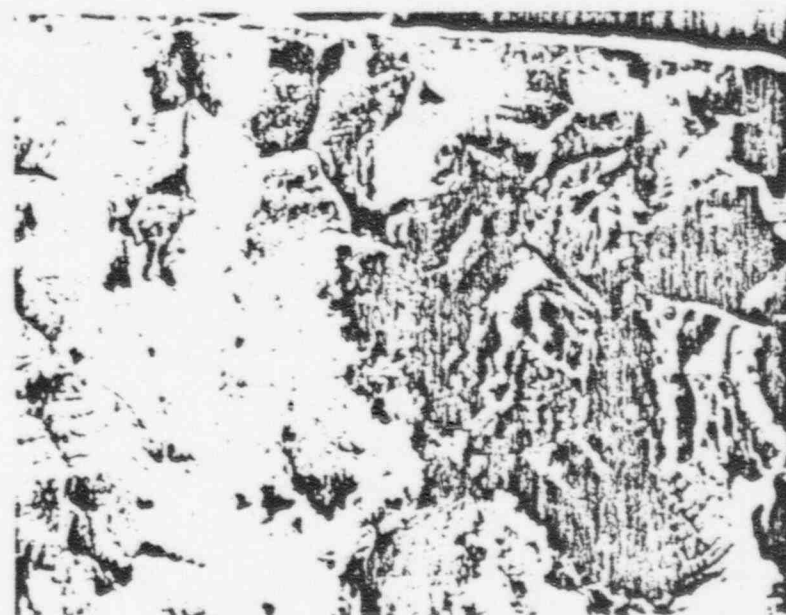


FIG. 8—IGSCC in sensitized (500°C/24 h) Type 304 stainless steel (Heat 812292) tested by dynamic strain in 289°C water with 8-ppm  $\text{O}_2$  at extension rate of 0.0008 mm/min.

of the heat-treat furnace. Apparently, the precipitation of chromium carbides occurs extremely fast during cooling from the solution-annealing temperature, such that a "seeding" effect results which enhances sensitization during subsequent thermal treatments in the sensitization range [1].

Additional work was conducted to assess the three measurement methods after more severe sensitization heat treatments. Measurements were performed on a heat of 2.5-cm (1-in.) plate (Heat X14902) which was sensitized 1, 4, 20, and 40 h at 620°C and furnace cooled. These results are given in Table 5, where all three methods indicate a saturation effect for this heat after sensitizing for 20 h or greater. All three methods reveal the lack of sensitization in the as-received condition (mill annealed). The EPR and A 262, Practice A, tests indicate a lower degree for specimens sensitized 4 h compared to 1 h, but this trend could be due to specimen variability, since the A 262, Practice E, tests were conducted on separate specimens while the A 262, Practice A, and EPR tests were conducted on the same specimens. All these sensitized specimens were susceptible to IGSCC after constant load testing in the severe environment of 289°C water with 8-ppm dissolved oxygen [6].

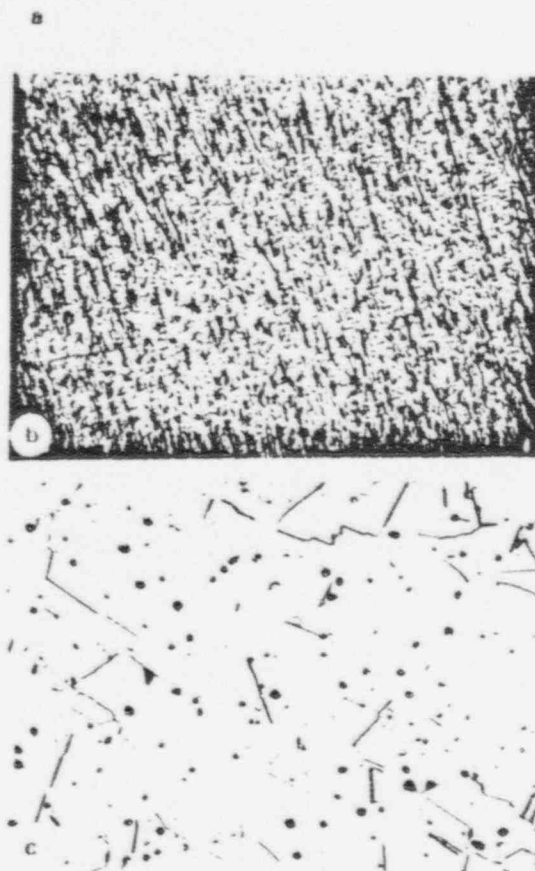
Six of the seamless pipe heats were evaluated further after sensitizing for 40 h at 620°C and furnace cooled. This sensitization treatment caused extensive IGSCC susceptibility in all the Type 304 stainless steel pipe spec-

POOR ORIGINAL

90014045

TABLE 5—Comparison of A 262, Practices A and E, with the EPR method for measuring degree of sensitization in Type 304 plate (Heat X14902).

Time at 620°C (1150°F) (h)	Average Penetration Rate mm/h (mil/h)	EPR Measurement $P_a$ (C/cm <sup>2</sup> )	Practice A (% Ditching)
As Received	0.00015 (0.006)	$< 10^{-1}$	$\leq 1$
1	0.0088 (0.345)	14.4	25 to 35
4	0.0232 (0.915)	11.1	3 to 5
20	0.0478 (1.88)	38.4	100
40	0.0432 (1.70)	39.5	100



POOR ORIGINAL

90014046

FIG. 9—Etch structures in Type 304 stainless steel forged bar (Heat 812292) sensitized 24 h at 500°C and tested for degree of sensitization using three techniques: (a) A 262, Practice E (0.1 to 0.4 mil penetration),  $\times 175$ . (b) EPR ( $P_a = 10.8 \text{ C/cm}^2$ ), (c) A 262, Practice A ( $\leq 1$  percent ditching),  $\times 175$ .

imens tested by dynamic strain or constant load in the highly aggressive 289°C water with 8-ppm oxygen [6]. A typical example is shown in Fig. 10 for Heat M7616 tested by dynamic strain. These results (Table 6) indicate that the A 262, Practice E, test is the most discriminating for greater degrees of sensitization. Both the EPR and A 262, Practice A, tests reflect a "saturation" effect (Fig. 11), where any additional sensitization is not

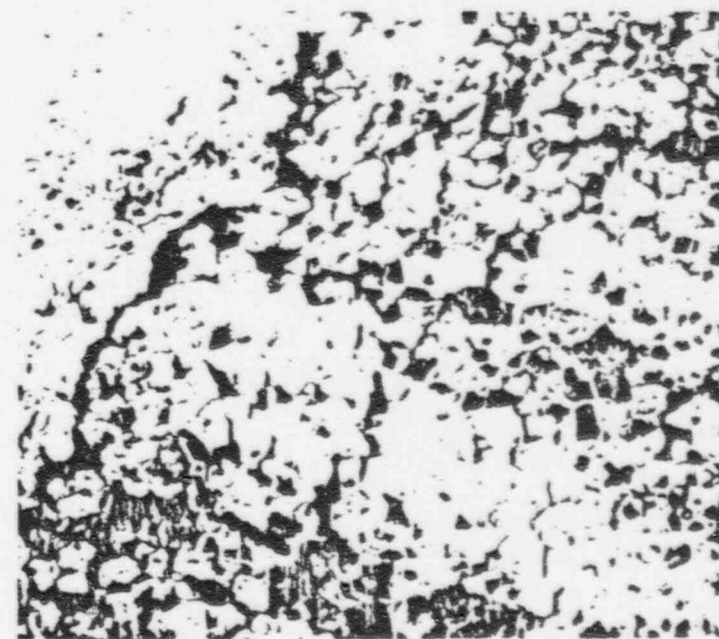
FIG. 10—Fracture surface of furnace-sensitized (620°C/40 h) Type 304 stainless steel (Heat M7616) tested by dynamic strain in 289°C water with 8-ppm O<sub>2</sub> at extension rate of 0.0008 mm/min.

TABLE 6—Comparison of three methods for measuring degree of sensitization of six IGSCC susceptible heats of Type 304 stainless steel seamless pipe sensitized 40 h at 620°C (1150°F).

Heat	A262-A (% Ditching)	A262-E mm/h (mil/h)	EPR, $P_a$ (C/cm <sup>2</sup> )
M7616	100	0.057 (2.23)	120
M7772	100	0.028 (1.11)	85
2P6424	100	0.017 (0.65)	77
2P6396	100	0.010 (0.40)	79
454659	100	0.002 (0.08)	100
TH6656	100	0.0005 (0.02)	85

distinguished by the test. The A 262, Practice A, test reveals a 100 percent ditched structure for all heats given a severe sensitization treatment. The EPR test produces very high  $P_a$  values but not a great deal of difference on a heat-to-heat basis, compared to that measured for the lower levels of sensitization.

In contrast to this, the quantified A 262, Practice E, test does appear to distinguish between heats given severe sensitization heat treatments since it is measuring a penetration mechanism, rather than a surface effect where a response is limited to the width of the affected grain boundaries.

### Discussion

The three test methods evaluated measure degree of sensitization in somewhat different manners [2]. The EPR method measures the current flow associated with active dissolution of chromium-depleted grain boundary areas as the potential is swept through the active range at a fixed rate. The A 262, Practice A, test evaluates the degree of grain boundary "ditching," presumably as a result of dissolution of grain boundary chromium carbides. Finally, the A 262, Practice E, test considers the extent of grain boundary attack which occurs in chromium-depleted areas in an aggressive corrodent at a fixed potential set by the solution. Each method has a set of "boundary" conditions within which it is capable of discriminating between different degrees of sensitization.

The upper boundary limit of the EPR test is the current associated with complete grain boundary activation. Once this condition is reached, the test will not be sensitive to higher degrees of chromium depletion. The lower limit is established by the sensitivity of the electronic circuitry for detection of current flow through the specimen.

The upper limit for A 262, Practice A, is 100 percent grain boundary ditching, after which no further discriminations between more severely

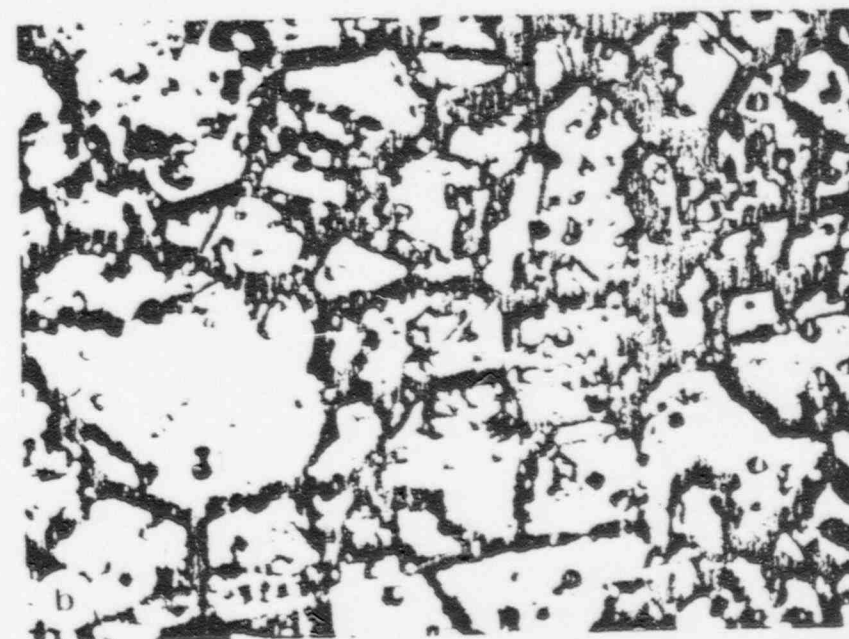
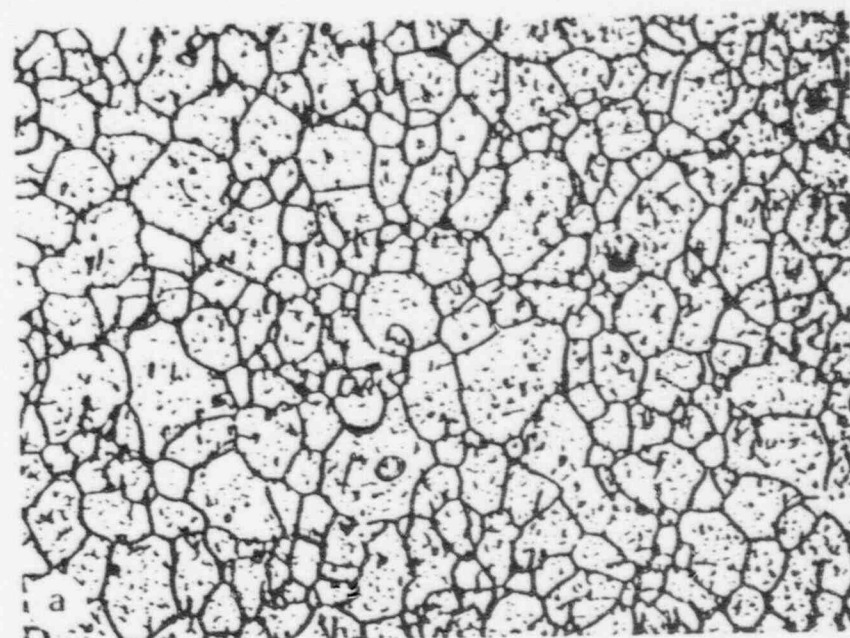


FIG. 11—Furnace-sensitized (620°C, 40 h) Type 304 stainless steel (Heat M7616) showing etch structures (a) after EPR testing,  $\times 118$ , (b) after A 262, Practice A testing,  $\times 200$ .

POOR ORIGINAL

90014047

sensitized materials can be made. No nationally recognized lower limit has been established for this test; below some degree of sensitization, no discriminations can be made because no grain boundary ditching is observed. It must be pointed up, however, that, according to ASTM procedures, it is not the objective of the A 262, Practice A, test to give quantitative data, but to provide qualitative results on a "go/no go" basis.

In the quantified A 262, Practice E, test there probably exists some upper limit of sensitization detection which is reaction rate limited, that is, the corrodent cannot reach the susceptible grain boundaries at a sufficiently rapid rate. However, this upper limit has not been observed in our tests to date. In the conventional A 262, Practice E, test, the upper limit is the observance of some degree of fissuring and would probably vary from one individual evaluation to another [4]. The lower limit for the quantified A 262, Practice E, test appears to be a penetration of about  $5.0 \times 10^{-4}$  cm (0.0002 in.). The exposure time required to achieve this depth of attack will vary with the degree of sensitization of the material (low levels of sensitization such as encountered in welding usually require at least three 72-h exposures). In the conventional A 262, Practice E, method, the lower limit is the lack of observance of "fissuring."

Given these characteristics of the three tests, one would expect the EPR test to be the most discriminating over the full range of degrees of sensitization and certainly the most useful for the levels of sensitization which are of principal concern to the industry. Although it is true the EPR test saturates out compared to the A 262, Practice E, method at higher degrees of sensitization (Fig. 12), these higher degrees are not of practical importance to the industry. The primary concerns relative to sensitization in Type 304 stainless steels are the low levels encountered during production and fabrication (for example, welding and heat treating). It is at these low levels of sensitization that the A 262, Practice E, test loses its discriminating power. Additionally, because of its limited range of detection, the A 262,

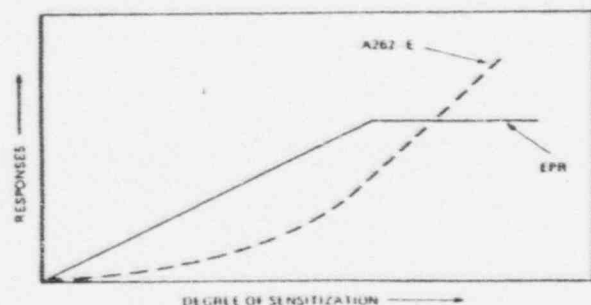


FIG. 12—Conceptual schematic of relative test behavior.

Practice A, test would be the least discriminating of the three methods over the full range of sensitization, and this hypothesis is supported by the results of this study and other performed at the General Electric Company [4].

A further advantage of the EPR technique is that it appears capable of providing "go/no go" indications of relative IGSCC susceptibilities for multiple heats of Type 304 stainless steel in moderately sensitized conditions. For example, a plot of  $I_{DS}$  versus EPR determined degree of sensitization ( $P_s$ ) for eleven of the twelve heats studied is shown in Fig. 13. Here, a least squares fit of the data provides a correlation coefficient of

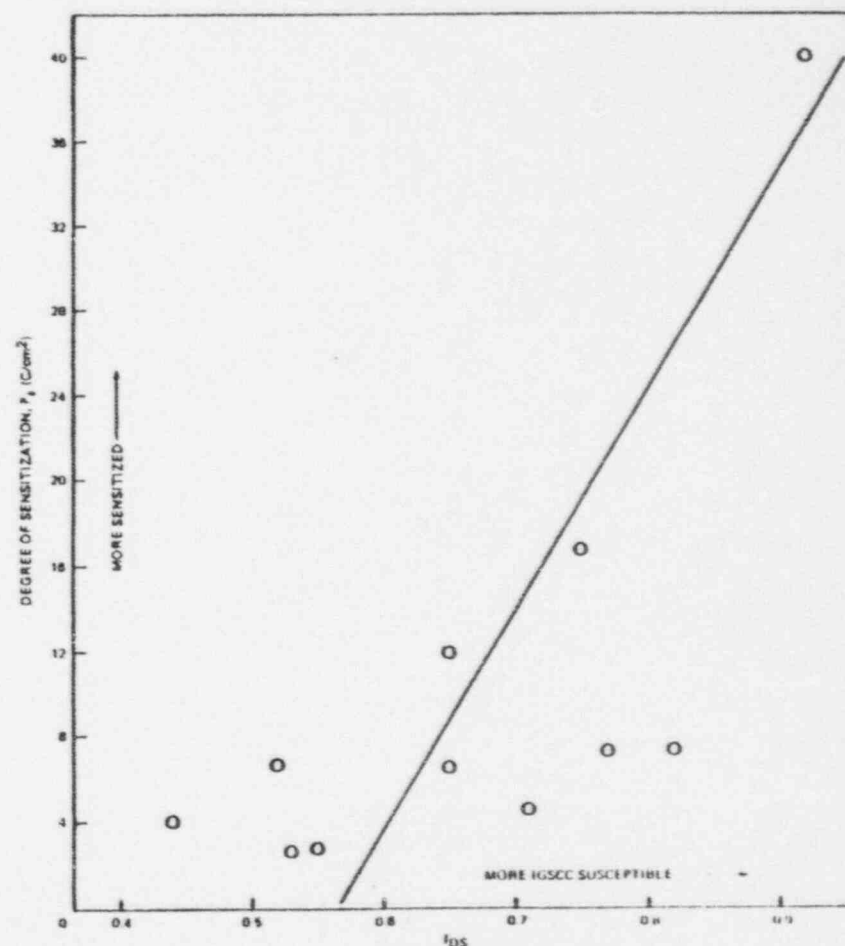


FIG. 13—Correlation between degree of sensitization and IGSCC resistance for eleven heats of Type 304 stainless steel sensitized 24 h at 500°C (correlation coefficient:  $r = 0.90$ ).



0.70, which indicates a good agreement between IGSCC in the extremely severe test used in this study and degree of sensitization after a moderate sensitization treatment. An even better correlation in this extremely aggressive environment is obtained by performing a similar analysis, but using welded specimens of the ten piping heats, where the degree of sensitization is slightly higher [5]. As shown in Fig. 14, the correlation coefficient here is 0.89, which significantly supports the potential of the EPR method for detecting IGSCC in sensitized stainless steel. Finally, work being conducted under U.S. Nuclear Regulatory Commission sponsorship [6] indicates that the EPR technique may be adapted for quantitative measurement of degree of sensitization in the field nondestructively, which is not possible using either of the other two measurement techniques.

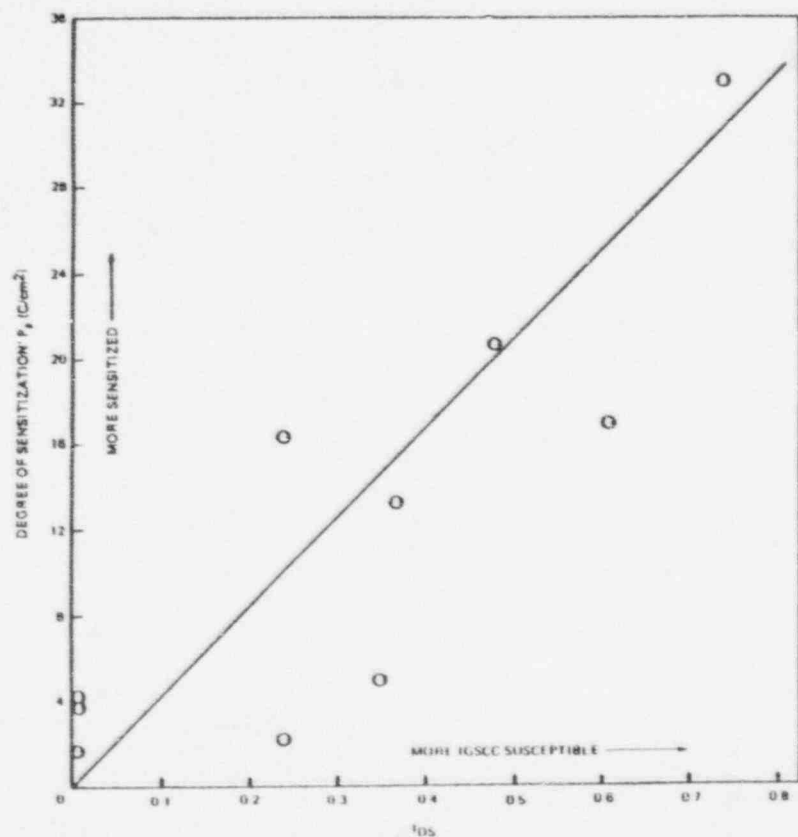


FIG. 14—Correlation between degree of sensitization determined by EPR and IGSCC resistance for ten heats of Type 304 stainless steel in as-welded condition (correlation coefficient,  $r_s = 0.89$ ).

## Conclusion

Based on the results of this study, the following conclusions can be made:

1. The EPR method is considered the most sensitive for quantitatively measuring the levels of sensitization which are of primary concern to the industry.
2. All three methods are capable of detecting moderate-to-severe degrees of sensitization in austenitic stainless steel.
3. Both the EPR and the A 262, Practice A, method appear to saturate at the higher degree of sensitization, which results in a loss of discriminating power between different heats of material.
4. The A 262, Practice E, test does not saturate, and retains its discriminating power at high degrees of sensitization, but it is not a suitable method for detection at the lower degrees of sensitization.
5. The EPR method is the most suitable for detecting potential susceptibility to IGSCC and is the only technique considered adaptable for obtaining quantitative information in the field nondestructively. For use in industry, this method will be required to correlate the EPR method to the industrial environment of concern if it is to be used as an indication of IGSCC potential rather than as a measure of sensitization caused by grain boundary chromium depletion.

## Acknowledgments

The authors gratefully acknowledge the assistance of V. M. Romero who conducted all the EPR measurements during this investigation. We are also indebted to G. E. Dunning for the A 262, Practice A results, to D. K. Blair for the A 262, Practice E testing, and to G. L. Smith for his very capable laboratory assistance in obtaining the IGSCC susceptibility data. We wish to thank the U.S. Nuclear Regulatory Commission and the Electric Power Research Institute for sponsoring much of this work. We would also like to express our appreciation to M. J. Povitch for his helpful discussions.

## APPENDIX I

### Determination of Carbide Precipitation in Wrought Austenitic Stainless Steels

#### Scope

This document describes a method for determining the relative amounts of carbide precipitation in the grain boundaries of wrought Types 304, 304L, 316, and 316L austenitic stainless steel used in this study.

### Applicable Documents, Codes, and Standards

**Codes and Standards**—The following codes and standards form a part of this specification to the extent specified herein.

**American Society for Testing and Materials**—(a) ASTM A 262, and (b) ASTM, Estimating an Average Grain Size of Metals (E 112-74).

### Examination Requirements

The material shall be evaluated in accordance with ASTM A 262, Practice A, as modified by this specification. The low-carbon grades, Types 304L and 316L, shall be heat treated (sensitized) at 1250°F for 1 h before testing.

The evaluation shall include a relationship between the length of the ditched grain boundaries and the total length of the grain boundaries, expressed as percentage, from a representative photomicrograph. The procedure shall consist of determining the ASTM grain size number per ASTM E 112. A representative photomicrograph at an appropriate magnification shall be prepared based on the following table and the portion of the grain boundaries that are ditched shall be crosshatched to facilitate measurement and calculation of relative percentage of ditched to total grain boundary (twin boundaries excluded).

Range of ASTM Grain Size Numbers	Photomicrograph Magnification
0 to 4.5	100
5.0 to 5.5	200
6.0 to 7.5	400
8.00 to 10	500

**Method**—The following illustrates the method to be used in the evaluation. The relative percentages of ditched grain boundary length versus the total grain boundary length are determined as follows. An overlay is made of the total grain boundary structure.

The differentiation between grain and twin boundaries from photomicrographs can be difficult, especially at the lower percentages of ditching. The simultaneous use of the photomicrograph and the same area viewed through the microscope may be necessary to distinguish between the grain and twin boundaries. Also, a second etching step may be used to delineate the grain boundaries. The second etching procedure consists of electrolytic 60 percent nitric acid ( $\text{HNO}_3$ ) at 40 mA/cm<sup>2</sup> and an etching time of 30 s. The time can be increased depending on the individual specimen requirements.

An alternate method for developing an overlay of the total grain boundary structure is to take a second specimen of material in close proximity and representing the same cross section and grain size. Grain boundary enhancement may be accomplished by either (a) sensitizing the specimen at 649 to 670°C (1200 to 1250°F), mounting, polishing, and etching with electrolytic oxalic acid, or (b) mounting, polishing, and etching with electrolytic  $\text{HNO}_3$  per the previous paragraph. The total grain boundary length may be taken from this overlay per the previous paragraph.

The portion of the total grain boundaries that are ditched should be identified

on the overlay. Discontinuous globular carbides are excluded from consideration as ditches.

The lengths of the total and ditched grain boundaries can be measured using an instrument, such as a Dietzgen "Plan Measure," Model 1719B, and the relative percentage of ditched to total calculated.

**Specimen Configuration**—The full cross section of material 2.5 cm (1 in.) thick or less shall be mounted to provide good edge retention. Thicker specimens may be cut and mounted separately so that the full cross section is examined.

### Quality Assurance Provisions

**Sampling**—Unless otherwise approved in an engineering-approved quality plan, at least one test specimen per each heat-treat lot shall be tested. If the heat-treat lot contains material from more than one mill heat, a separate specimen from each mill heat shall be tested. Forgings, pipe, and other product forms shall be obtained with sufficient additional material to make test specimens.

**Certificate of Test**—For vendor-supplied material, a statement of test results shall be included in the material test certificate which accompanies the material or shipment and shall include the measured percentage of ditched grain boundary. In addition, a photomicrograph used for the evaluation properly marked with the heat number and heat-treat lot number shall be provided.

## APPENDIX II

### Quantitative Measurement of General Intergranular Corrosion Susceptibility

#### Scope

This standard establishes a method for the quantitative determination of the susceptibility of metals to general intergranular corrosion in aggressive chemical environments.

This test method is intended to standardize laboratory test procedures and to provide a means of quantifying the susceptibility of both homogeneously heat-treated materials and materials with localized areas of differential sensitization (such as weld heat-affected zones) to intergranular corrosion.

The method described here is directed primarily toward austenitic alloys which sensitize by carbide precipitation and associated chromium depletion of the grain boundary material. However, the general method may be used with any metal or alloy in any condition by selection of the appropriate test solution.

#### Summary of Method

This method represents a modification of ASTM A 262, Practice E. The ultimate tensile strength of the material of interest in the desired heat-treat condition is determined and compared with the apparent ultimate tensile strength of corrosion-tested material. The difference between these two values is used to calculate the corrosion which occurred during the test period. The accuracy of measurement of specimen gage section dimensions and failure load becomes increasingly important as the corrosion rate decreases. As described here, the method results in a mean

the baseline specimen should be pulled first. If more than one baseline specimen is provided for a set, the baseline specimens should be distributed through the set to account for any drift in the tensile machine load cell.

The load cell used for the tests should have the smallest full-scale load range which will accommodate all the specimens to minimize errors in interpolation of failure load. A cell with a full-scale value of 454 kg (1000 lb) is recommended, with specimen gage cross-sectional area adjusted to this limit as described in the second paragraph under the Specimen Preparation heading.

Recorder charts of the load curve for each specimen shall be provided with the failed specimens. Interpolation of the failure load shall be made to the nearest pound. Accurate interpolation is critical when corrosion rates are low.

The failed halves of each specimen shall be taped together securely and identified externally with the specimen number.

All specimens to be evaluated metallographically shall be subjected to 1 to 3 percent strain at room temperature with the surface of interest in tension. Higher strains may lead to tearing of the unattacked material and misleading results. Specimens shall be polished to a depth sufficient to remove edge effects from straining.

#### Corrosion Rate Calculations

The baseline failure stress shall be calculated for each of the baseline specimens for a common set and averaged to give mean baseline failure stress.

The corrosion rate shall be calculated for each individual corrosion-tested specimen and the mean corrosion rate and standard deviation calculated for each specimen group. This calculation is performed on the basis of the change in apparent failure stress of the corrosion tested specimens as follows

$$\text{corrosion rate} = \frac{r_o - \sqrt{\frac{\sigma_f}{\pi \sigma_u}}}{\text{exposure hours}}$$

where

$r_o$  = original specimen radius,  
 $\sigma_f$  = failure load, and  
 $\sigma_u$  = ultimate tensile strength.

On specimens evaluated metallographically, the depth of attack may be measured directly with a calculated eyepiece (Filar) or from photomicrographs of the surface. At least  $\times 100$  is recommended, and higher magnifications may be required for shallow attack. The corrosion rate is calculated by dividing the depth of penetration (in centimetres) by the exposure time (in hours). Representative photographic documentation is recommended.

#### Sample Evaluation

The fractured tensile difference specimens shall be examined visually and optically for indications of unexpected conditions and all such results noted on the data sheet. These include observations, such as nonuniform necking, preferential intergranular attack, failure in the weld rather than the weld heat-affected zone, and failure in the gage shoulder rather than the gage section. In addition, the location and nature

of the failure should be compared between specimens from the same set to evaluate the physical consistency of the results.

Location of the maximum depth of penetration, in reference to the weld center line, should be recorded for each specimen. Differences in depth of penetration between the two sides of the heat-affected zone should be noted if present. Differences in depth of penetration between the surface of primary interest and the opposite surface should be noted. Photographic documentation of observed differences is recommended.

## APPENDIX III

### Electrochemical Potentiokinetic Reactivation (EPR) Method for Determining Degree of Sensitization in Stainless Steels

#### Scope

This appendix describes an EPR test method for quantitatively determining the degree of sensitization in thermally treated AISI Types 304, 304L, 316, and 316L stainless steels.

#### Applicable Documents, Codes, and Standards

**Codes and Standards**—The following codes and standards form a part of this specification to the extent specified herein: (a) ASTM E 112-74, (b) ASTM Recommended Practice for Standard Reference Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements (G 5-72), (c) ASTM Recommended Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing (G 3-74), and (d) ASTM Preparation of Metallographic Specimens (E 3-62).

#### Equipment

**Standard Polarization Cell**—Requirements shall be in accordance with Section 2.1 of ASTM G 5.

**Electrode Holder**—Requirements shall be in accordance with Section 2.2 of ASTM G 5.

**Potentiostat**—Requirements shall be in accordance with Section 2.3 of ASTM G 5.

**Potential Measuring Instruments**—Requirements shall be in accordance with Section 2.4 of ASTM G 5.

**Current Measuring Instruments**—Requirements shall be in accordance with Section 2.5 of ASTM G 5.

**Current Integration Measurement Instruments**—An instrument that is capable of integrating the current under the curve developed during reactivation should be used. The instrument should be capable of measuring a current integral (in coulombs) to within an accuracy of 1 percent of the absolute value of a current range between 0.001 and 1000 mA.

**Anodic Polarization Circuit**—Requirements shall be in accordance with Section 2.6 of ASTM G 5, except the current integration instrument should be inserted in series with the lead between the working electrode and potentiostat.

**Electrodes**—Working electrodes can be any shaped piece of metal at least 0.32 cm (1/8 in.) in diameter or on a side, by any suitable thickness, which has a stainless steel electrode holder mounting screw spot-welded on the side opposite to where the measurement will be taken and is potted in a suitable innocuous compound such that only one planar surface is exposed to the electrolyte.

**Counter Electrodes**—Requirements are in accordance with Section 2.7.2 of ASTM G 5.

**Calomel Reference Electrode**—Requirements are in accordance with Section 2.8 of ASTM G 5.

### Standard Experimental Procedures

**Test Specimen Preparation**—Prepare the surface within 1 h of the experiment or store the prepared specimen in a suitable desiccating cabinet. Wet grind with 240-grit and 400-grit SiC paper, followed by wet polish with 600-grit silicon carbide (SiC) paper until previous coarse scratches are removed, rinse with water, and dry. The specimens should be final polished in two additional stages with 6 and 1  $\mu$ m diamond paste or 0.05- $\mu$ m alumina slurry on a nylon or silk cloth over microcloth prepared polishing wheel per ASTM E 3.

Mount the specimen on the electrode holder as described in Section 2.2.1 of ASTM G 5. Tighten the assembly by holding the upper end of the mounting rod in a vise or clamp while tightening the mounting nut until the gasket is properly compressed.

Clean the specimen just before immersion into the electrolyte by degreasing with a suitable detergent, rinsing in distilled water then alcohol, and air drying.

Prepare 1 litre of 0.5 M  $H_2SO_4$  + 0.01 M potassium thiocyanate (KCNS) from reagent grade chemicals and distilled water (solution can be made up in bulk and stored for one month). Transfer approximately 500 to 600 ml of solution to clean polarization cell.

Bring the temperature of the solution to  $30 \pm 1^\circ C$  by immersing the cell in a controlled temperature water bath or by other convenient means.

Place the specimen, platinum auxiliary electrodes, salt bridge probe, and other components in the test cell. Ensure that the salt bridge is filled with the test solution and contains no air bubbles, particularly in the restricted space within the tip region. The levels of the solution in the reference and polarization cells should be the same to avoid siphoning.

Purge the solution before test initiation for about 2 min and continuously during the test with high purity nitrogen gas (99.90 percent minimum) at 150 cm<sup>3</sup>/min.

Adjust the salt bridge probe tip so it is as close to the specimen surface as possible, but not touching the sample or mount.

Record the open-circuit (rest) specimen potential, that is, the corrosion potential, after about a 2-min immersion. If the rest potential does not register normal for the class of alloys being evaluated (–350 to –450 mV for Type 304 stainless steel), then cathodically charge the specimen at –600 mV for 2 to 5 min and recheck the rest potential. If the rest potential is still abnormal (usually around –200 mV), the specimen must be removed from the flask and repolished to eliminate the tarnish film.

Passivation is accomplished by setting the potential to +200 mV versus standard calomel electrode and holding for 2 min. Complete passivation can be checked by observing the lack of change on the current integrator instrument output.

**Reactivation Scan**—Start the potential backscan (cathodic direction) using a potentiodynamic sweep rate of 6 V/h ( $\pm 5$  percent).

Rezero and start the current integrator instrument, recording the current continuously with change in potential.

The recorder automatically plots the anodic polarization data on semilogarithmic paper in accordance with ASTM G 3. It is acceptable for the EPR evaluation to plot with the potential as the abscissa and the current as the ordinate, in opposition to the recommended standard reference plot in ASTM G 3.

Lock reading on current integrator when current is close to the initial corrosion potential and has just reversed polarity. Record this reading as the integrated current value (in coulombs) in data record sheet.

Put all electrochemical polarization equipment on standby and remove *E* versus *i* plot from recorder for inclusion in data file.

Remove specimen from cell and holder, rinse in distilled water followed by alcohol rinse, then air dry.

### Data Acquisition and Analysis

Test parameters should be recorded as follows: EPR Run No., Specimen No., Material, Heat; Surface Condition; Specimen Location; Test Temperature, Sweep Rate; Passivating Potential/Time; Rest Potential. Use the following data record sheet form or equivalent for recording these data.

Test data to be recorded include the charge, *Q*, in coulombs (integrated current under anodic portion of curve during reactivation) maximum anodic curve peak height in milliamperes, and Flade potential in millivolts (potential at which anodic curve breaks upward during reactivation). Record on data record sheet.

Each potted specimen should be photographed after test (without additional preparation or etching) at suitable magnification to document the microstructures and extent of grain boundary grooving after the EPR test. An additional photomicrograph must be taken at  $\times 100$  to measure grain size. If the specimen is not sufficiently etched after the EPR test to delineate the microstructure for grain size determination, then the specimens should be etched with 10 percent oxalic acid, electrolytically as per ASTM A 262, Practice A, and a photomicrograph obtained. Attach photos to data record sheet.

Determine the surface area by measuring all dimensions to the nearest 0.1 mm.

The integral charge value, *Q*, should be normalized to the grain boundary area (GBA) of each specimen using the relationship

$$P_a(C/cm^2) = \frac{Q}{GBA}$$

and

$$Q = \text{charge measured on current integrating instrument (coulombs)},$$

$$GBA = A_s [5.09544 \times 10^{-3} \exp(0.34696 X)],$$

where

$$A_s = \text{specimen Area (cm}^2\text{) and}$$

$$X = \text{ASTM grain size at } \times 100.$$

Note: Show calculation in data record sheet.

### Electrochemical Potentiokinetic Reactivation—Data Record Sheet

1. EPR run number \_\_\_\_\_
2. Specimen number \_\_\_\_\_

3. Material and product form \_\_\_\_\_
4. Material heat number \_\_\_\_\_ Heat treat lot no. \_\_\_\_\_
5. Specimen location \_\_\_\_\_
6. Specimen surface area,  $A_s$  \_\_\_\_\_  $\text{cm}^2$
7. Initial rest (corrosion) potential \_\_\_\_\_ mV versus saturated calomel electrode (SCE)
8. Test temperature \_\_\_\_\_  $^{\circ}\text{C}$
9. Passivating potential time \_\_\_\_\_ mV versus SCE/time
10. Sweep rate \_\_\_\_\_ V/h
11. Integrated current,  $Q$ , \_\_\_\_\_ coulombs
12. Maximum anodic current \_\_\_\_\_ mA
13. Flade potential \_\_\_\_\_ mV versus SCE
14. Grain size,  $X$  \_\_\_\_\_ ASTM grain size
15. Normalized charge per grain boundary area ( $P_g$ ) calculation

GBA = grain boundary area,

GBA =  $A_s [5.09544 \times 10^{-1} \exp. (0.34696X)]$

GBA = \_\_\_\_\_

where

$A_s$  = Specimen area in  $\text{cm}^2$ ,

$X$  = ASTM grain size at  $\times 100$ ,

$P_g$  =  $Q/\text{GBA}$  where  $Q$  = integrated current in coulombs, and

$P_g$  = \_\_\_\_\_

16. Attach:
  1. Micrograph characterizing microstructure and extent of grain boundary grooving.
  2. Separate micrograph, if needed, used to determine grain size.

Date: \_\_\_\_\_

Determined by: \_\_\_\_\_

## References

- [1] Cowan, R. L. and Gordon, G. M., "Intergranular Stress Corrosion Cracking and Grain Boundary Composition of Fe-Ni-Cr Alloys," *Proceedings, International Conference on Stress Corrosion Cracking and Hydrogen Embrittlement of Iron Base Alloys*, Unieux-Firminy, France, June 1973.
- [2] Cowan, R. L. and Tedmon, C. S., "Intergranular Corrosion of Iron-Nickel-Chromium Alloys," *Advances Corrosion Science and Technology*, Plenum Press, New York, 1972.
- [3] Clarke, W. L., Cowan, R. L., and Danko, J. C. in *Stress Corrosion Cracking—the Slow Rate Technique*, ASTM STP 665, American Society for Testing and Materials, 1979.
- [4] Walker, W. L., "A Comparison of Several Methods Used to Measure the Degree of Sensitization of Type 304 Stainless Steel," General Electric Report NEDO-13342, July 1973.
- [5] Clarke, W. L., Romero, V. M., and Danko, J. C., "Detection of Sensitization in Stainless Steel Using Electrochemical Techniques," NACE Corrosion Conference, Paper No. 180, San Francisco, Calif., March 1977.
- [6] General Electric Report GEAP 21382, Aug. 1976, U.S. Nuclear Regulatory Commission NUREG-0251-1, available from National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Va. 22161.

POOR ORIGINAL

90014053