ANL/ET/CP-99988

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December 1999

Manuscript to be presented at Corrosion 2000, Orlando, FL, March 26-31, 2000.

*Work supported by the U.S. Nuclear Regulatory Commission, Office of Nuclear Regulatory Research.

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ABSTRACT

Certain safety-related core internal structural components of light water reactors, usually fabricated from Type 304 or 316 austenitic stainless steels (SSs), accumulate very high levels of irradiation damage (20-100 displacement per atom or dpa) by the end of life. Our data bases and mechanistic understanding of the degradation of such highly irradiated components, however, are not well established. A key question is the nature of irradiation-assisted intergranular cracking at very high dose, i.e., is it purely mechanical failure or is it stress-corrosion cracking? In this work, hot-cell tests and microstructural characterization were performed on Type 304 SS from the hexagonal fuel can of the decommissioned EBR-II reactor after irradiation to ≈50 dpa at ≈370°C. Slow-strain-rate tensile tests were conducted at 289°C in air and in water at several levels of electrochemical potential (ECP), and microstructural characteristics were analyzed by scanning and transmission electron microscopies. The material deformed significantly by twinning and exhibited surprisingly high ductility in air, but was susceptible to severe intergranular stress corrosion cracking (IGSCC) at high ECP. Low levels of dissolved O and ECP were effective in suppressing the susceptibility of the heavily irradiated material to IGSCC, indicating that the stress corrosion process associated with irradiation-induced grain-boundary Cr depletion, rather than purely mechanical separation of grain boundaries, plays the dominant role. However, although IGSCC was suppressed, the material was susceptible to dislocation channeling at low ECP, and this susceptibility led to poor work-hardening capability and low ductility.

Key words: core internals, stainless steel, irradiation damage, intergranular stress corrosion cracking, electrochemical potential, grain-boundary Cr depletion, dislocation channeling

INTRODUCTION

As neutron fluence increases, austenitic stainless steel (SS) core internals of boiling and pressurized water reactors (BWRs and PWRs) become susceptible to irradiation-assisted intergranular (IG) cracking. Although most failed components can be repaired or replaced, such operations are difficult and very expensive. Therefore, extensive research has been conducted in the last ≈20 years to develop our understanding of this form of degradation.[1-34]

For BWR or BWR-like conditions at relatively low damage levels (e.g., ≈1-10 dpa), irradiation-induced grain-boundary depletion of Cr has been considered by most investigators to be the primary metallurgical process that causes irradiation-assisted stress corrosion cracking (IASCC). Very narrow Cr-depleted zones at grain boundaries have been observed by field-emission-gun analytical electron microscopy[12,13,18,19] and Auger electron spectroscopy.[10,13,14,28] Furthermore, the results of electrochemical potentiokinetic reactivation tests[5] and the effects of electrochemical potential (ECP)[8,10] and dissolved oxygen (DO)[9,10] on susceptibility of nonirradiated, thermally sensitized material (where Cr depletion is widely recognized as the primary factor) to intergranular stress corrosion cracking (IGSCC) and on susceptibility of BWR-irradiated solution-annealed material to IASCC during slow-strain-rate tensile (SSRT) tests[8-10] are reported to be similar.

However, contrary to expectations based on the strong effect of ECP on SCC associated with grain-boundary Cr depletion, IG cracking of highly stressed components has been reported in PWRs[1,2,20] (which operate at low ECPs), and the susceptibility of PWR-irradiated components[4] or specimens[29] to IG cracking at low ECP has been demonstrated in hot cell experiments.[4,29] In the experiment of Manahan et al.,[4] 10%-cold-worked Type 304 SS specimens were tested in Ar at ≈315°C after irradiation to ≈7 dpa in a PWR. Although the authors considered that these specimens contained IG-type brittle-fracture morphology on as much as ≈35% of the fracture surfaces, actual high-magnification fractographs do not indicate that they are true IG separation, especially when the fracture surface morphology is compared with clear IG cracking in control rod cladding in PWR water.[2,4] However, clear IG cracking in Ar gas or low-DO water has been reported by Hide et al.[27] for thermally sensitized Type 304 SS specimens irradiated to ≈0.4 dpa in water at ≈290°C in the Japan Material Testing Reactor and tested at ≈290°C. The IG crack morphology was limited, however, to a small fraction (<5%) of the fracture surface near the specimen free surface, which should contain a high concentration of O (due to corrosion during service).

If we consider this background, there appears to be no real evidence for the occurrence of clear IG cracking in inert gas or air in solution-annealed austenitic SSs at temperatures relevant to light water reactors (LWRs), at least for Type 304 SS irradiated up to ≈7 dpa. However, as irradiation damage is increased to very high levels (i.e., 20-100 dpa), very significant microstructural evolution occurs, e.g., extensive Cr depletion and extensive segregation of Ni, Si, and other impurities on grain boundaries, and formation of dense defect clusters, microvoids, and irradiation-induced precipitates in grain matrices. The result is that properties at very high dose, such as hardening, grain matrix deformation, grain-boundary amorphization, mechanical IG cracking, and susceptibility to IGSCC, could differ significantly from those of steels that have been exposed to relatively low doses of irradiation. Information on these properties helps us to better understand the susceptibility of safety-significant core internals (such as BWR top guide and core plate and PWR baffle bolts) to failure at the end of life. To this end, Type 304 SS specimens that had been fabricated from the hexagonal fuel can of the decommissioned EBR-II reactor after irradiation to ≈50 dpa at ≈370°C, were tested in air and water, and posttest analyses were performed by scanning and transmission electron microscopies (SEM and TEM) to elucidate the failure mechanism(s) at very high doses. Initial tests were conducted at ≈289°C, whereas tests at ≈325°C in PWR-like water will follow.

MATERIALS AND EXPERIMENTAL PROCEDURES

The geometry of the specimens for tensile and SSRT tests is shown in Fig. 1. The specimens (nominal wall thickness 1 mm, or 0.040 in.) were machined in a hot cell from the hexagonal fuel can of the EBR-II reactor, which was decommissioned after more than 30 years of operation. The fuel can, fabricated from a commercial-grade solution-annealed Type 304 SS, was irradiated in liquid Na at ≈370°C to a fluence of ≈1.02×10^{23} n $\cdot cm^{-2}$ (E > 0.1 MeV), which corresponds to a damage level of ≈50 dpa. The average grain size of the as-irradiated specimens was ≈35 µm. No records of the composition of the archive ingot or the as-fabricated fuel can were available. The machined irradiated specimens were ground and polished to remove burrs and surface irregularities, and then, the final width and wall thickness of the specimen gauge section were measured at three locations before testing.

Tensile properties and susceptibility to IG cracking were determined by SSRT tests on the specimens at 289°C in air and in water at a strain rate of 2.5×10^{-7} s^{-1}. All water tests were conducted at 289°C in deionized high-purity water that contained ≈8 or ≈0.01 ppm DO. Concentration of DO, controlled by purging the deaerated water with an N_2/O_2 mixture, was measured on the effluent side. Conductivity and pH of the water at room temperature were in the range of ≈0.061-0.067 µS cm^{-1} and 6.7-7.1, respectively. Electrochemical potential was measured at the effluent side at regular interval. Further details of the SSRT test procedure are reported in Ref. 26.

Posttest fractographic analysis was conducted by SEM to measure the percentage of ductile, transgranular, and IG fracture surface morphologies. One or two disks were carefully cut out of the fracture tips (adjacent to the fracture surface) of the cracked specimen. Thin-foil TEM specimens were jet-polished at room temperature in a solution of 25-mL perchloric acid, 225-mL acetic acid, and 50-mL butylcellosolve. TEM analysis was performed at 100 keV in a JEOL 100-CXII scanning transmission electron microscope.

RESULTS

Figure 2 shows engineering stress vs. elongation of specimens that were tested in air and water. Feedwater chemistry (i.e., DO, ECP, conductivity, and pH) and results from SSRT tests (i.e., 0.2%-offset yield strength, maximum strength, uniform strain, and total strain) are summarized in Table 1. Also shown in the table are the results of SEM fractographic analysis (i.e., ductile, IG, and transgranular fracture surface morphology) of the failed specimens.

Deformation and Failure Behavior in Air

The highly irradiated steel exhibited good work-hardening capability and surprisingly high ductility in air, which is manifested by uniform and total elongations as high as ≈3.5 and ≈4.8%, respectively. Tensile properties of the ≈50-dpa material are shown in Fig. 3, with similar data reported for commercial-grade Type 304 SSs that were irradiated to <5 dpa and tested in BWR-like conditions. Strength of the EBR-II material at ≈50 dpa was significantly lower than that of BWR components at <5 dpa that were tested under similar conditions, i.e., ≈680 vs. ≈850 MPa in yield strength and ≈780 vs. 900 MPa in ultimate tensile strength. This finding is most likely due to the fact that irradiation temperature of the former material was significantly higher than that of the latter (i.e., ≈290 vs. 370°C).

Fracture surface morphology of the air-tested specimen was entirely ductile (see Fig. 4); no evidence of IG separation was observed in any part of the fracture surface. Many dislocation loops and microvoids were observed in the material (Fig. 5). However, there was no evidence that microvoids, typically ≈20-60 nm in size, aggregated on grain boundaries. This finding is consistent with the observation that the material did not fail by IG separation in air.

Results of TEM characterization of the thin-foil specimen, cut out of the gauge section adjacent to the fracture surface, showed that twinning was the predominant deformation mechanism (see Fig. 6). Twinned grains exhibited characteristic twin reflections in selected area diffraction patterns (SADs), and clear dark-field images of (111) twins could be obtained by using the twin reflections (Fig. 6).

Failure Behavior in High-ECP Water

In contrast to the deformation behavior in air, in an oxidizing environment of the high-ECP water (ECP +202 mV SHE, DO ≈8 ppm), the material exhibited negligible work-hardening capability and poor ductility, as evidenced by low uniform and total elongations of only ≈0.5 and ≈2.4%, respectively. Deformation steps on specimen side surfaces were absent. However, as indicated by IG fracture surface morphology as high as ≈90% in one test and brittle cracking near the shoulder-region hole in another, the material exhibited high susceptibility to IGSCC (see Fig. 7).

Failure Behavior in Low-ECP Water

In the low-ECP water (ECP -318 mV SHE, DO ≈0.01 ppm), the material exhibited low work-hardening capability and poor ductility (uniform and total elongations ≈0.7 and ≈2.1%, respectively). However, despite the poor ductility, percent IGSCC was negligible, and the fracture surface of the specimen was essentially ductile (see Fig. 8). No evidence of IG separation was observed, showing that low levels of DO and ECP were effective in suppressing the susceptibility of the heavily irradiated material to IGSCC. The free side surface of the fracture tip was characterized by high-density deformation steps, indicating that localized deformation occurred in the low-ECP water (Fig. 8).

Despite negligible susceptibility to IGSCC, ductility of the material was significantly lower in low-ECP water (-318 mV SHE) than in air (i.e., total elongation 2.1 vs. 4.8%). Because this behavior indicates that differing types of deformation mechanisms operate in air and in low-ECP water, TEM analysis was conducted on disk specimens that were carefully excised from the fracture tips of the specimens. The primary deformation mode in the low-ECP water was dislocation channeling (see Fig. 9). This is in contrast to the observation that the primary deformation mode of the material in air was twinning. In the low-ECP water, twinning was negligible. Twins and dislocation channels exhibit diffraction behavior and dark-field-imaging characteristics that differ distinctively.

In addition to dense dislocation loops and microvoids, the highly irradiated material contained dense irradiation-induced precipitates. These dense precipitates were present in all specimens, showing that they did not precipitate during the tests. Dark-field images showed that short line dislocations were frequently "decorated" with precipitates. Although the precipitates could not be identified conclusively at this time, they were cleared in dislocation channels (Fig. 9). Grain-boundary offsets were also observed (see Fig. 9B).

DISCUSSION

Results from these experiments show that despite the very high dose level, IG failure did not occur in the EBR-II-irradiated steel in air or in low-ECP water. However, similar to Type 304 SS components or specimens irradiated to ≈2-5 dpa in BWRs, the EBR-II-irradiated steel exhibited extensive susceptibility to IGSCC in high-ECP oxidizing water. Furthermore, as shown in Fig. 10, the effects of ECP and DO on the susceptibility of the ≈50-dpa material to

IGSCC are very similar to those of Type 304 SS BWR core internal components. This observation indicates that the stress corrosion process associated with irradiation-induced grain-boundary Cr depletion plays the primary role in BWR-like oxidizing water.

Obviously, the results of the present experiment do not support the premise that purely mechanical separation of grain boundaries occurs or is likely to occur in water at very high irradiation doses. The results also indicate that if sufficiently low levels of ECP can be maintained, susceptibility to IASCC could be suppressed by use of proper water chemistry even at the end of life, at least in BWR-like conditions at ≈290°C.

It is not clear why dislocation channeling is promoted, and, at the same time, twinning is suppressed in low-ECP water. It is likely that as suggested by Bruemmer et al., twins are nucleated only when stress in a grain matrix is sufficiently high and reaches a critical stress.[15] If dislocation channeling is promoted by some mechanism in low-ECP water, then strain hardening will be limited to the dislocation channels or to the immediate vicinity of the channels; hence, it will be more difficult for a grain matrix as a whole to reach the critical stress. This premise, then, implies that at very high levels of irradiation some factor related to the presence of low-ECP water (e.g., hydrogen uptake or vacancy generation) promotes dislocation channeling. Furthermore, the observation that the fine irradiation-induced precipitates were cleared in dislocation channels indicates that dense irradiation-induced precipitation plays an important role in dislocation channeling.

CONCLUSIONS

Slow-strain-rate tests at 289°C and posttest microstructural examination were conducted on material from a Type 304 stainless steel (SS) hexagonal fuel can that was irradiated to ≈50 dpa at ≈370°C in the EBR-II reactor. Although the irradiation conditions are not completely prototypical, the material would represent a limiting end–of–life fluence for boiling-water reactor (BWR) internal components. Major observations and findings are as follows:

1. No intergranular failures were observed in tests in air at 289°C. This suggests that intergranular failures cannot occur by purely mechanical processes at <50 dpa.

2. As in the case of BWR internal components irradiated to 2–5 dpa, intergranular failures were not observed in tests in water at low electrochemical potentials. However, virtually complete intergranular failure was observed in tests in water at high electrochemical potentials. These results are consistent with the premise that irradiation-induced grain-boundary Cr depletion plays a major role in irradiation-assisted stress corrosion cracking.

3. In the tests in air, extensive twinning leads to relatively high tensile ductilities in Type 304 SS at 289°C even at ≈50 dpa.

4. In low-electrochemical-potential water at 289°C, tensile ductilities were low in spite of the fact that intergranular failures were not observed. The twinning observed in the air tests was negligible, and dislocation channeling was the primary process for deformation and failure in the low-electrochemical-potential water. Dense irradiation-induced precipitation and the presence of the low-electrochemical-potential water appear to play important roles in dislocation channeling.

ACKNOWLEDGMENTS

The authors thank M. B. McNeil for many helpful discussions, C. L. Trybus for providing the slow-strain-rate tensile specimens, and L. J. Nowicki for preparing the specimens for SEM and TEM analyses. This work was supported by the U.S. Nuclear Regulatory Commission, Office of Nuclear Regulatory Research.

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TABLE 1
SSRT TEST RESULTS FOR TYPE 304 STAINLESS STEEL SPECIMENS[a] FROM THE DECOMMISSIONED EBR-II REACTOR HEXAGONAL FUEL CAN[b]

			Feedwater Chemistry				SSRT Parameters				
Specimen ID No.	α−γ Hot Cell ID No.	SSRT Test[c] No.	Oxygen Conc. (ppm)	Average ECP (mV SHE)	Conductivity at 25°C ($\mu S \cdot cm^{-1}$)	pH at 25°C	Yield Stress (MPa)	Max. Stress (MPa)	Uniform Strain (%)	Total Strain (%)	Fracture Surface Morphology
A-1	521-A	IR-96-10		tested	in air		680	776	3.54	4.82	ductile
E-1	521-C	IR-96-9	8.0	+210	0.061	6.87	specimen	fractured	across the	shoulder	-
B-1	521-B	IR-96-11	8.0	+202	0.067	7.10	447	472	0.47	2.41	90% IGSCC
F-1	521-D	IR-96-12	0.01	-318	0.065	6.71	602	664	0.68	2.12	channel fracture

[a]Specimen size 44.5 x 12.7 x 1.0 mm, gage section 12.7 x 3.2 x 1.0 mm
[b]Discharged after irradiation to ≈50 dpa (fluence 1.1 x 10^{23} n cm^{-2}, E > 0.1 MeV) in sodium at ≈370°C
[c]Test at 289°C at a strain rate of 2.5 x 10^{-7} s^{-1} in deionized high-purity water or air

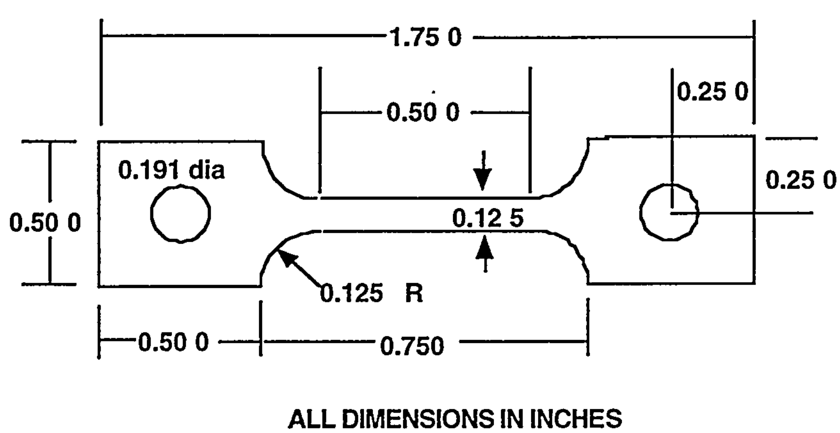


FIGURE 1: Geometry of SSRT test specimens prepared from EBR-II reactor hexagonal fuel can.

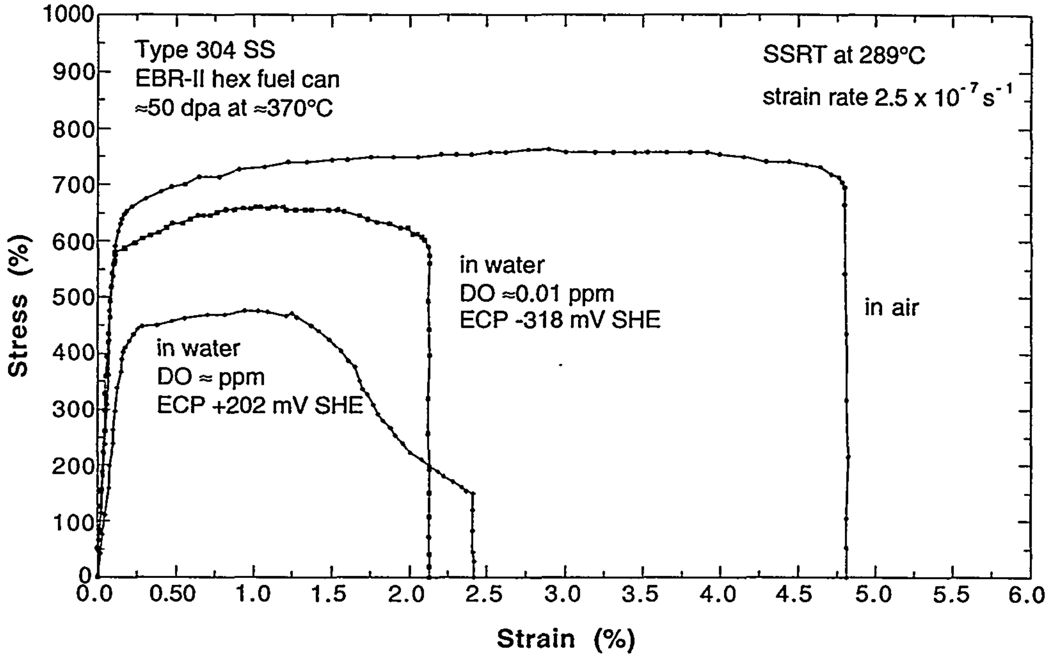


FIGURE 2:
Engineering stress vs. strain of Type 304 SS from hexagonal fuel assembly can irradiated in EBR-II to ≈50 dpa at ≈370°C and tested at 289°C in air and high-purity water.

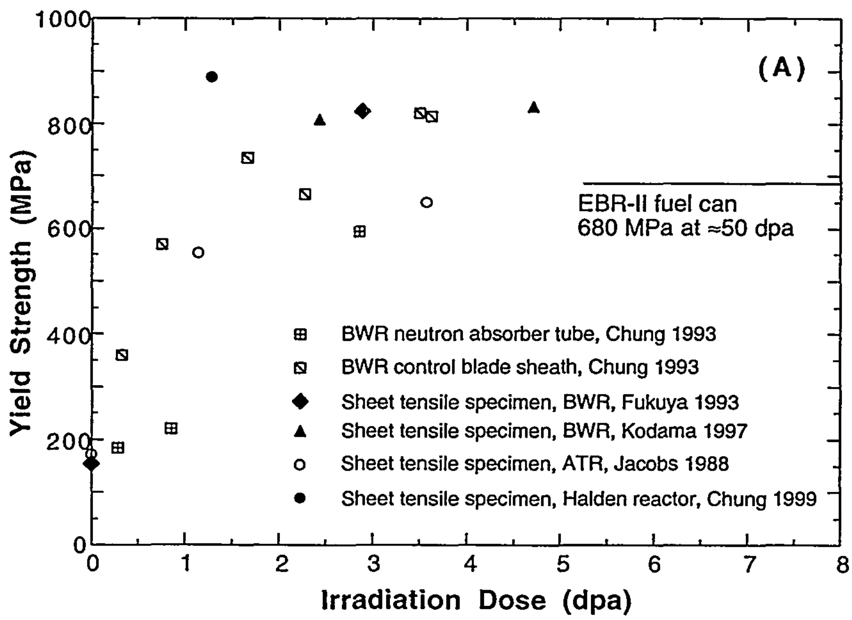
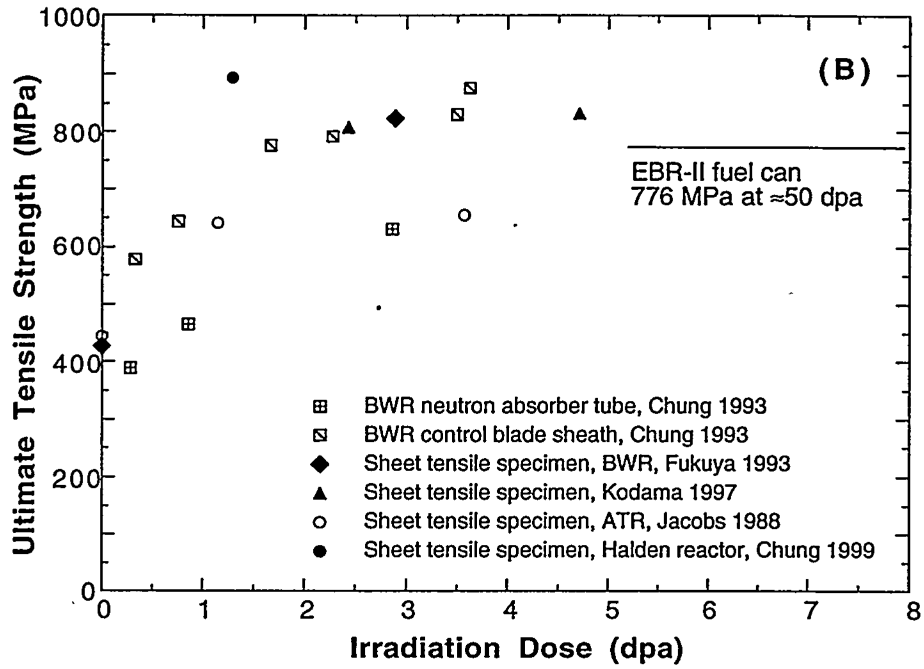


FIGURE 3:

Comparison of (A) yield strength, (B) ultimate tensile strength, and (C) total elongation of EBR-II fuel can, irradiated at ≈370°C to ≈50 dpa and tested in air at 289°C, with similar data for solution-annealed commercial-grade Type 304 SS irradiated in BWR and tested at ≈289°C.



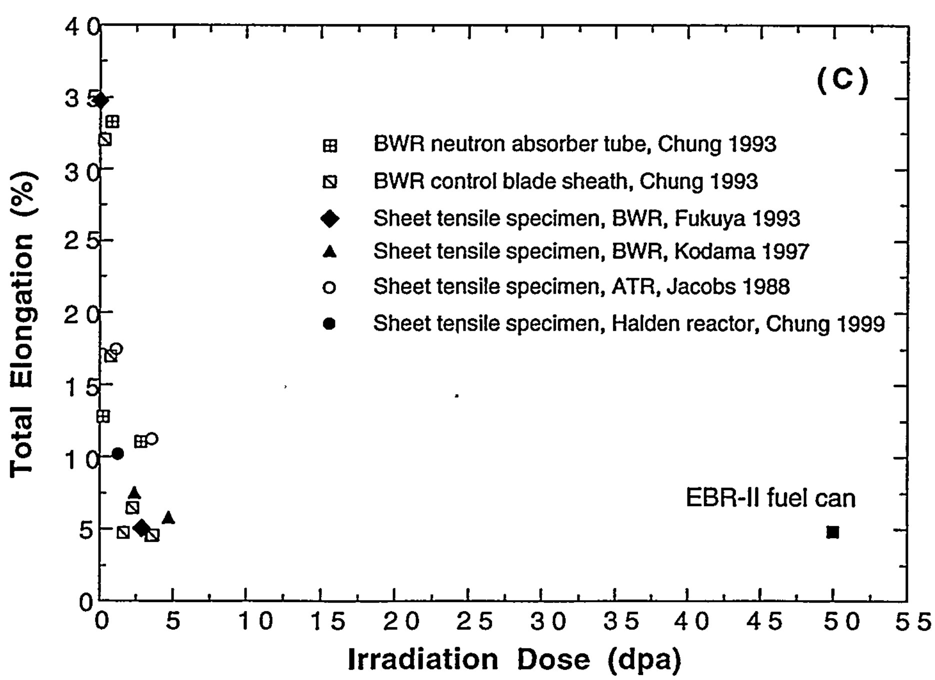


FIGURE 3: Continued.

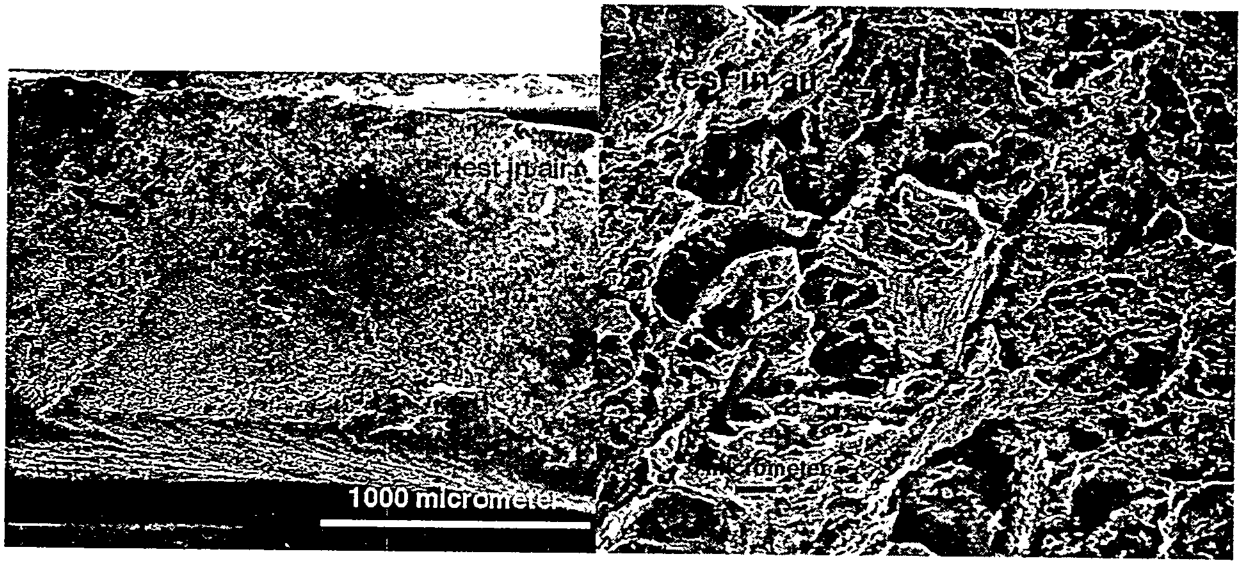


FIGURE 4:
Low- and high-magnification SEM fractographs of specimen tested in air.

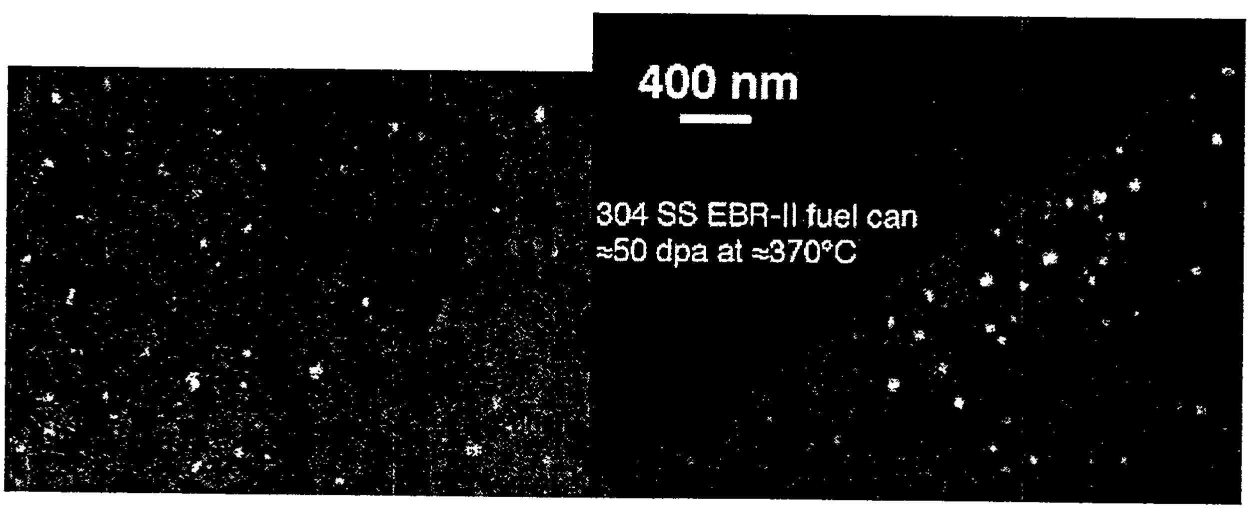


FIGURE 5:
Bright-field TEM images showing (left) dense dislocation loops and voids and (right) void distribution near grain boundary.

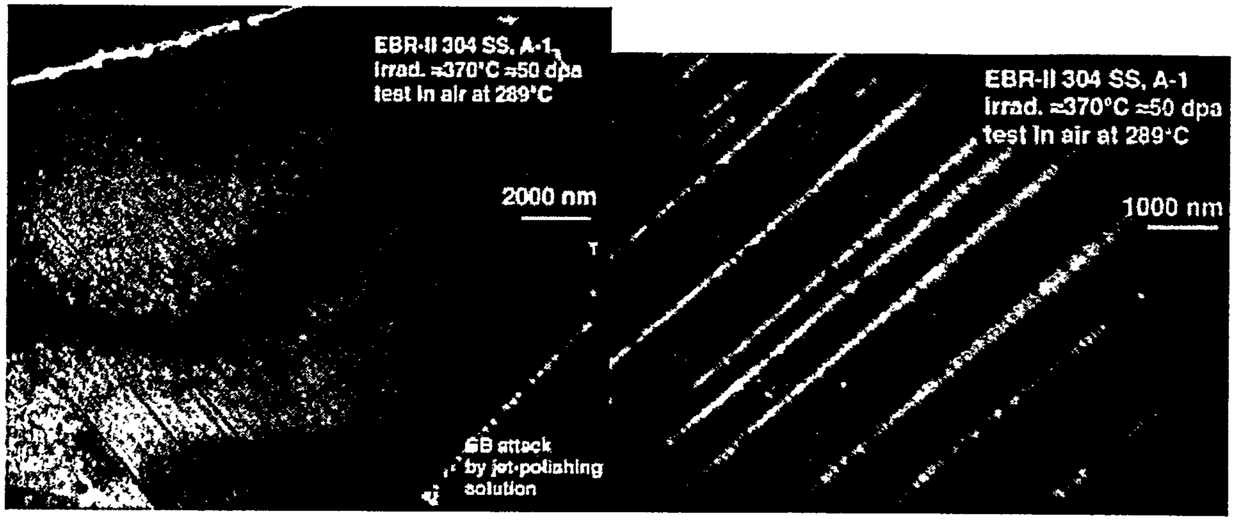


FIGURE 6:
Twins in fracture tip of specimen tested in air: (left) bright-field image and (right) dark-field imaged produced with a twin reflection.

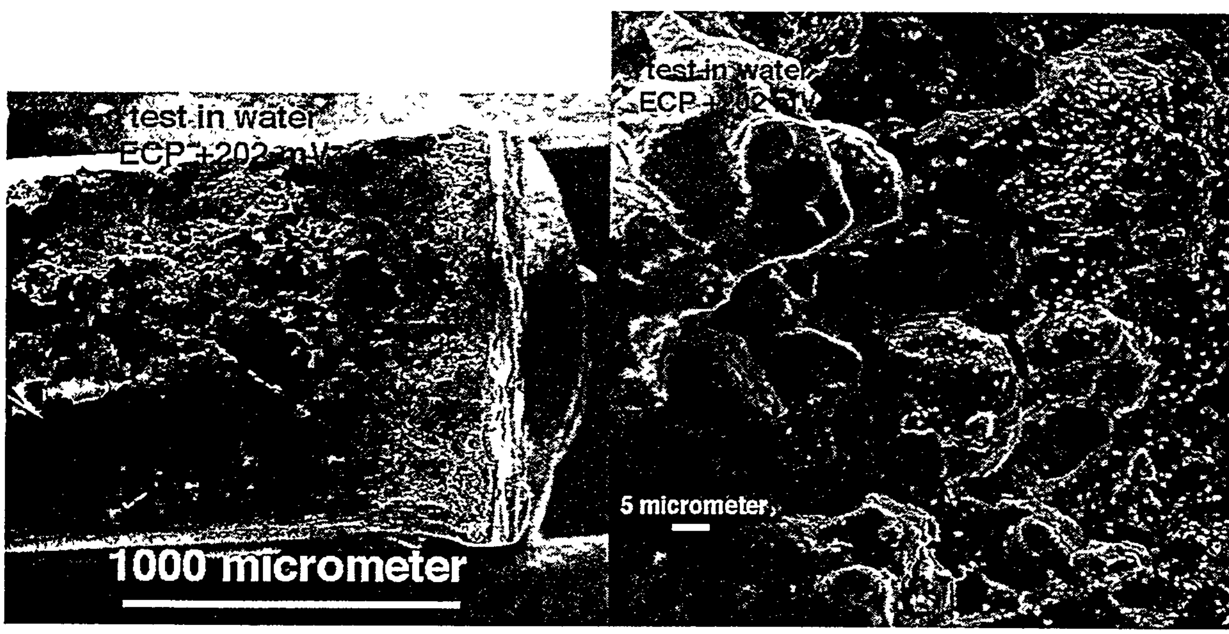


FIGURE 7:
Low- and high-magnification SEM fractographs of IG fracture in specimen tested in water, ECP +202 mV.

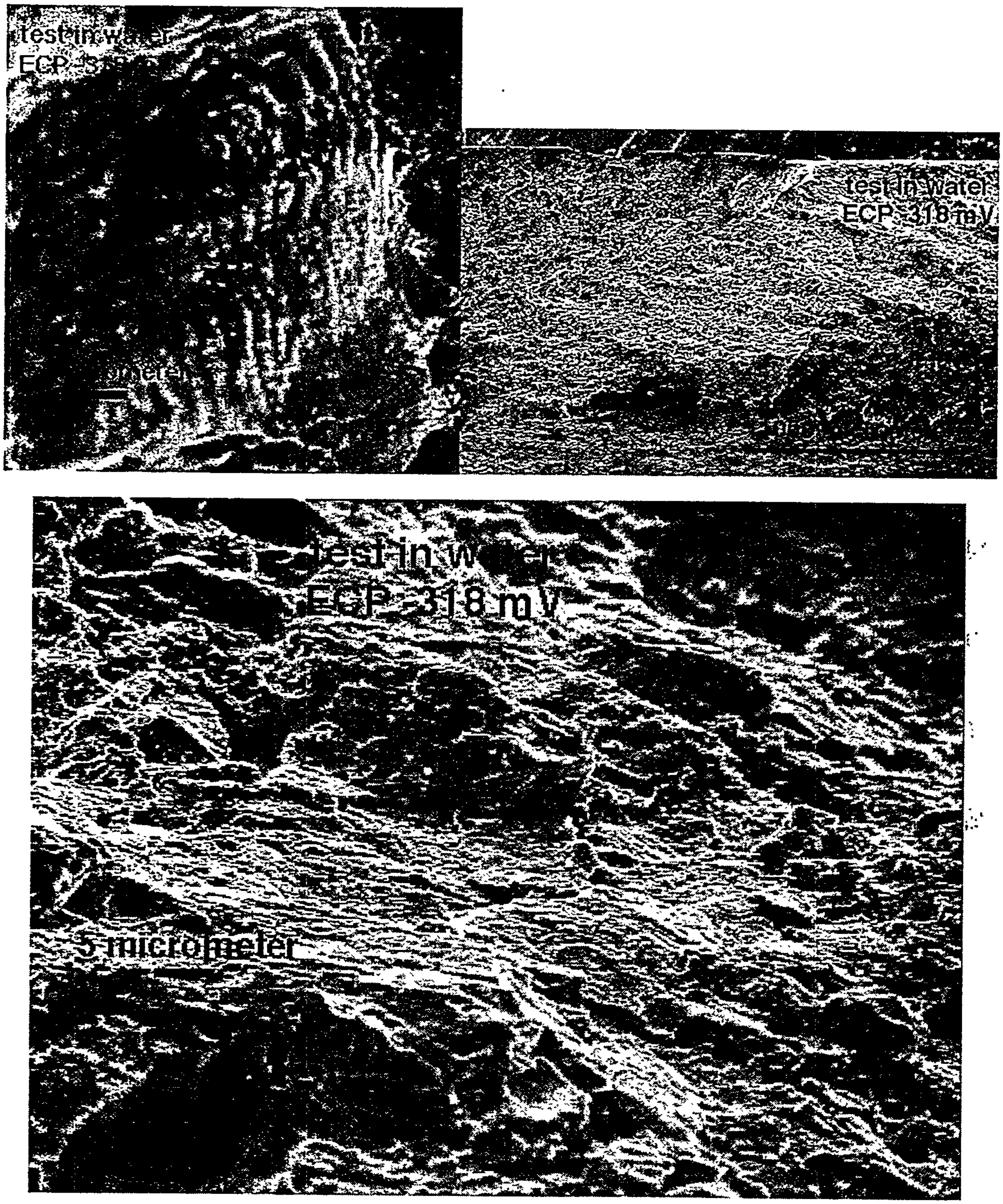
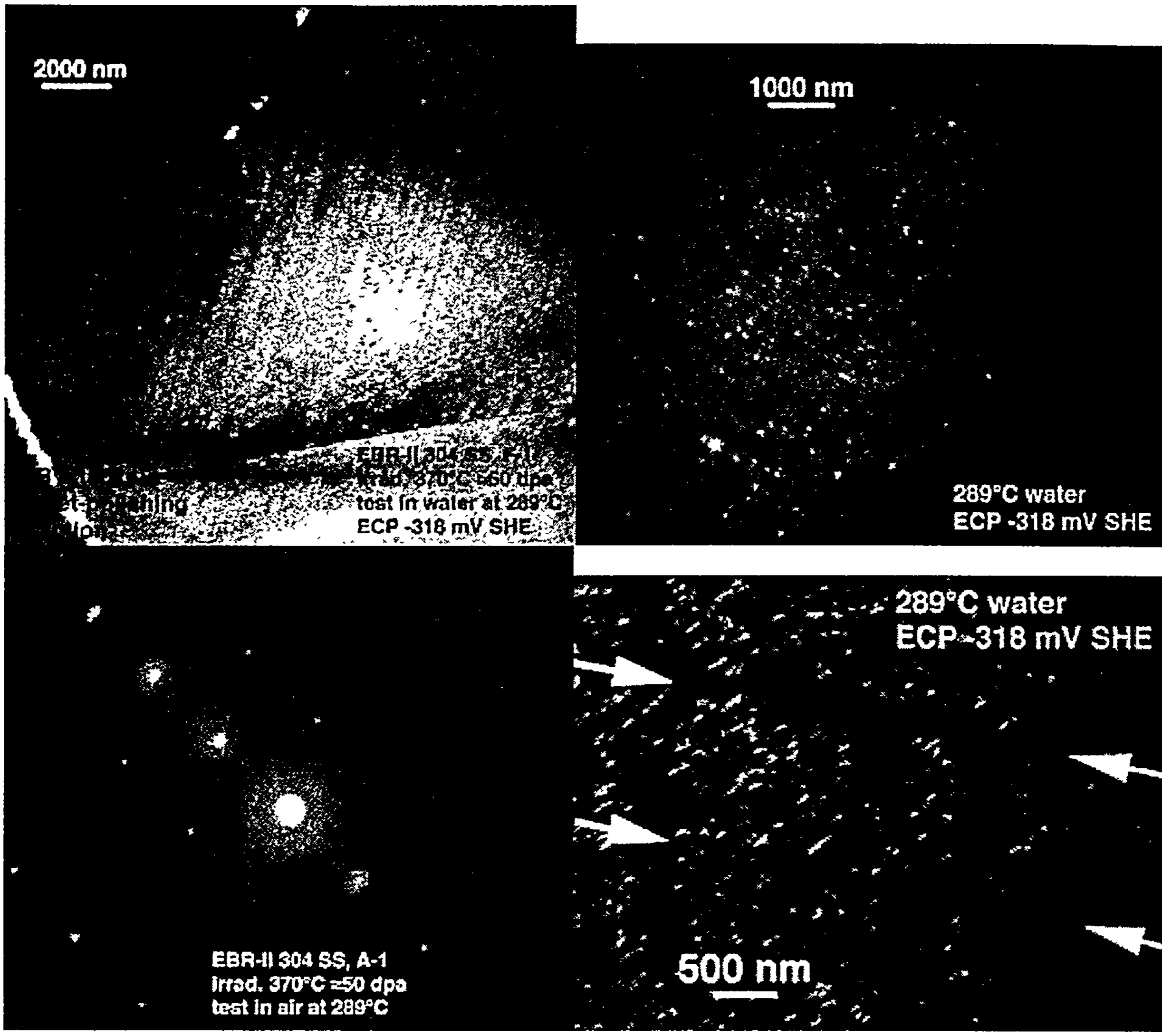


FIGURE 8:
SEM photomicrographs of specimen tested in water, ECP -318 mV: (top left) side surface of fracture tip showing deformation steps, (top right) fracture surface at low magnification, and (bottom) fracture surface at high magnification.



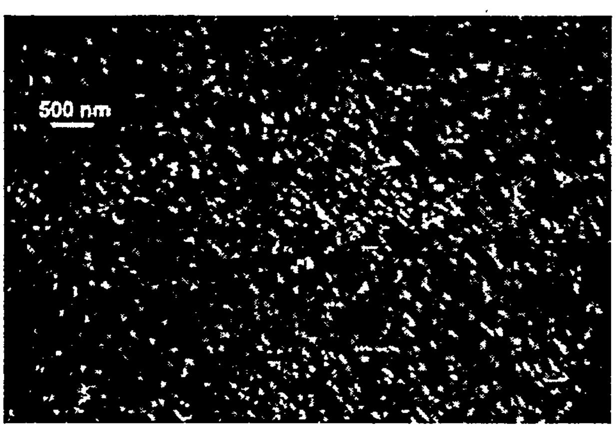


FIGURE 9:

Microstructure of the fracture tip of the specimen tested in water, ECP -318 mV: (top left) bright-field image showing dislocation channels and grain-boundary offset, (top right) high-magnification bright-field image of channels, (middle left) selected area diffraction pattern showing reflections from unidentified precipitates, (middle right) dark-field image from a precipitate reflection showing that dislocation channels are cleared of precipitates, and (bottom left) dark-field image showing that short line dislocations are decorated with precipitates.

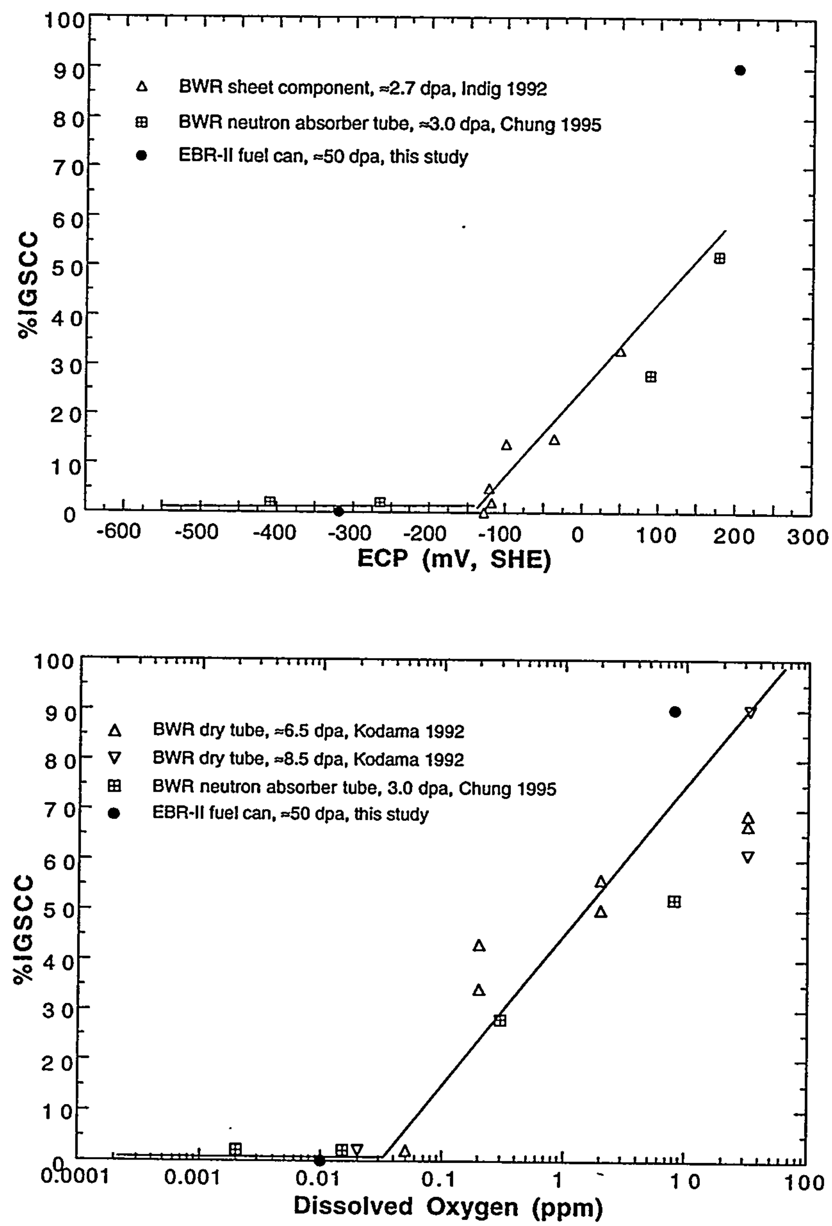


FIGURE 10:
Susceptibility to IGSCC (in percent IGSCC) vs. ECP (top) and DO (bottom) of commercial-grade Type 304 SS BWR components and EBR-II fuel can.