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RADIATION HARDENING AND RADIATION-
INDUCED CHROMIUM DEPLETION EFFECTS
ON INTERGRANULAR STRESS CORROSION
CRACKING OF AUSTENITIC STAINLESS STEELS

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RADIATION HARDENING AND RADIATION-INDUCED CHROMIUM DEPLETION EFFECTS ON INTERGRANULAR STRESS CORROSION CRACKING OF STAINLESS STEELS

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Abstract

Radiation hardening and radiation-induced chromium depletion are related to intergranular stress corrosion cracking (IGSCC) response among various stainless steels. Available data on neutron-irradiated materials have been analyzed and correlations developed between fluence, yield strength, grain boundary chromium concentration and cracking susceptibility in high-temperature water environments. Large heat-to-heat differences in the critical fluence (0.2 to 2.5 n/cm²) for IGSCC are documented. In many cases, this variability is consistent with yield strength differences among irradiated materials. IGSCC correlated better to yield strength than to fluence for most heats suggesting a possible role of the radiation-induced hardening (and microstructure) on cracking. However, isolated heats reveal a wide range of yield strengths from 450 to 800 MPa necessary to promote IGSCC which cannot be understood by strength effects alone. Grain boundary chromium depletion is found to qualitatively explain differences in IGSCC susceptibility for irradiated stainless steels. Examination of measured chromium contents versus SCC shows that all materials showing IG cracking have some grain boundary depletion ($\geq 2\%$). Grain boundary chromium concentrations for cracking (below ~ 16 wt%) are in good agreement with similar SCC tests on unirradiated 304 SS with controlled depletion profiles. Heats that prompt variability in the yield strength correlation, are accounted for by differences in their interfacial chromium contents. Certain stainless steels are more resistant to cracking even though they have significant radiation-induced chromium depletion. It is proposed that chromium depletion is required for IASCC, but observed susceptibility is modified by other microchemical and microstructural components.

Introduction

Irradiation-assisted stress corrosion cracking (IASCC) continues to be a concern in light-water reactor (LWR) core component materials. Although, research has increased as evidenced by the International Cooperative Group on IASCC,¹ there is no agreement how radiation exposure affects SCC susceptibility. Numerous interactions are possible between radiation and environment-induced cracking. Neutron irradiation is known to significantly alter material microstructure and microchemistry, accelerate kinetic processes, and produce changes in solution chemistry. These effects directly influence the local mechanics, reactivity and electrochemistry of the crack tip as illustrated in Figure 1. Recent reviews by Andresen^{2,3} present a good overview of the important issues involved in IASCC and

of the current understanding of individual material, mechanical and environmental effects on cracking susceptibility.

This paper examines two primary material aspects affected by irradiation exposure: radiation hardening and radiation-induced chromium depletion. Both aspects increase with irradiation fluence and may play a role in IASCC susceptibility. Available data from neutron-irradiation experiments have been compiled and analyzed to develop correlations between hardening, chromium depletion and SCC. Radiation hardening is quantified by measurements of yield strength, while primary depletion measurements are made by high-resolution analytical electron microscopy using a field-emission-gun, scanning transmission electron microscope (FEG-STEM). For the most part, IGSCC susceptibility has been assessed by post-irradiation, slow-strain-rate (SSR) tests on stainless steels in oxygen-containing (0.2-32 ppm), high-purity, 288°C water. Strain rates for the IGSCC tests used in the evaluation ranged from 1.6 to 3.7×10^{-7} /s.

Fluence Effects on IGSCC

A classic example illustrating IASCC is the SSR test results of Jacobs, et al.⁴ on BWR-irradiated, commercial purity (CP) 304 SSs. Intergranular cracking was first observed at a fluence of about 5×10^{20} n/cm², in good agreement with in-core, control blade sheath cracking.⁵ Percent IG cracking in the SSR tests (strain rate of 3.7×10^{-7} /s, 32 ppm O₂) was used to gage material susceptibility, and reached nearly 100% at a fluence of $\sim 3 \times 10^{21}$ n/cm². This data is shown in Figure 2(a) and (b) along with results from several other sources.⁶⁻¹⁰ Plotting fluence on a linear scale in 2(b) is done to expand the 304 SS data and allow direct comparison of SCC to other variables examined in following sections.

Kodama, et al.⁶ used an identical approach to Jacobs for evaluating IASCC in neutron-irradiated, CP 304 and 316 SS heats. Once again, IGSCC was observed to increase sharply above a critical fluence level. No significant IG cracking was detected at 1.2×10^{21} n/cm² and a fluence of $\sim 6 \times 10^{21}$ n/cm² was required to approach 100% IGSCC. Differences and similarities are documented in Figure 2 showing that the Kodama data (for O₂ contents from 2 to 32 ppm) appears to be simply shifted to higher fluences. An accurate fluence to promote IGSCC is difficult to define, but is about 2×10^{21} n/cm². This 'critical' fluence level is about 4 times that reported by Jacobs for 304 SS.

The SSR test results of Chung, et al.⁷ indicated susceptibility to IGSCC at a much lower fluence ($\leq 0.2 \times 10^{20}$ n/cm²) for a high-purity (HP) 304 SS absorber tube material. Although only 3 fluence levels were tested, a similar trend in cracking with increasing fluence is seen, but shifted to a lower fluence. The increased susceptibility is surprising since SSR tests were conducted in a less aggressive environment (0.3 ppm O₂). Identical SSR tests on an irradiated CP 304 SS absorber tube by Chung reveal a cracking response between that for Jacobs and Kodama in Figure 2. A second CP 304 SS (specimens removed from a control-blade sheath) has also been examined by Chung. In this case, very little IGSCC was observed even at fluences up to 2.5×10^{21} n/cm². Consistent with this heat-to-heat variability is the CP 304 SS data of Clarke and Jacobs⁹ and of Jacobs, et al.¹⁰ showing isolated samples resistant to IGSCC at fluences above 2×10^{21} n/cm² while similar specimens failed by nearly 100% IGSCC. The comparison between the data of Kodama and that of Jacobs for 316 heats also shows an apparent difference in susceptibility. These differences in IGSCC response suggest that the "critical" fluence for susceptibility may vary by more than an order of magnitude among stainless steel heats.

One cause for some of the variability in the collective data is an uncertainty in the reported fluence. Fluence needs to be converted into a term more representative of radiation-induced material damage. A first step in this direction is to convert fluence to displacements per atom (dpa). Unfortunately, this conversion is complex and requires detailed knowledge of the neutron energy/intensity spectrum which depends on the reactor type and sample position within the core. A typical value for stainless steels in a BWR is $\sim 0.7 \times 10^{21}$ n/cm² per dpa for neutron energies above 1 MeV.² Insufficient information is reported for these irradiations to convert n/cm² to dpa and assess effects on the IGSCC correlation. However, the advanced test reactor (ATR) irradiations are expected to be less effective in producing damage than LWR irradiations due to its faster neutron spectrum.

Radiation Hardening Effects on IGSCC

Limited work has been conducted characterizing microstructural evolution during LWR irradiation with the majority of analysis performed for higher temperature and higher dose conditions.¹¹ The primary microstructural change during low-temperature (288°C)/low-dose irradiation of stainless steels is the formation of small vacancy and interstitial loops. Loop densities and sizes increase with dose and Frank loops may unfault to form a dislocation network, significantly hardening the matrix. This prompts a large increase in yield strength, sharply decreases ductility and toughness, and leads to inhomogeneous deformation behavior.

Although direct comparisons have not been made between radiation-induced microstructural evolution and IGSCC, yield strength changes with fluence are related to the evolving defect microstructure.¹² Measurements^{4,6-9,13} have been obtained on various stainless steels as a function of irradiation dose. The 304 SS (BWR) data of Jacobs⁴ are the most complete and are representative of the heats examined for IGSCC. A rapid rise in yield strength is observed with increasing fluence to about 900 MPa at 3×10^{21} n/cm² (~ 4 dpa). ATR irradiations tend to show lower strengths that reach about 70% of the BWR data. Several differences among the data sets can be detected. Although strength increases with dose, the BWR-irradiated 304 SSs that cracked at lower fluences tend to have higher strengths. For example, the IGSCC-susceptible HP heat of Chung exhibits yield strengths more than double the more resistant CP absorber tube material.

Yield strength measurements are compared to IGSCC results in Figure 4. Nearly all of the BWR-irradiated materials of Jacobs (304 SS) and Kodama (304 and 316 SS) fall very close to one another. Initial cracking in the SSR tests is seen only after the yield strength has increased to about 600 MPa (more than 3 times the typical value for annealed stainless steel). In addition, the two absorber tube heats (HP and CP) of Chung are now consistent with one another, reflecting the much higher strength and IG cracking measured for the HP material. However, the IGSCC versus strength curve is shifted to lower strengths with initial cracking observed at ~ 450 MPa even though the SSR test environment is less aggressive. Interestingly, the ATR-irradiated heats (304 and 316 SS) also show cracking at lower strengths. One heat having very high strength and very little IGSCC is the CP 304 SS sheath material of Chung. A large difference in the yield strengths required to promote IGSCC is evident between the sheath (800 MPa) and absorber (450 MPa) materials.

Many of the differences among data sets in Figure 2 are accounted for by comparing to the material yield strength in Figure 4. This indicates that the yield strength is a better measure of radiation damage than fluence. It also suggests that the radiation-induced hardening and microstructure may be playing a role in IGSCC.

Hardening alone does not appear to be sufficient to explain cracking susceptibility, but may act in combination with radiation-induced segregation. The hardened matrix will localize plastic deformation in grain boundary regions at loads below the macroscopic yield stress. At plastic strains, transgranular deformation characteristics will also be localized on specific planes after an initial dislocation has cleared a channel through the impeding loops. Inhomogeneous deformation of this type has been recently documented by Gorynin, et al.¹⁴ in a stainless steel irradiated at 300°C to a fluence of 1×10^{21} n/cm² ($E > 1$ MeV). Detailed characterization of irradiation effects on interfacial deformation is needed along with direct comparisons between radiation-induced microstructural evolution and IGSCC susceptibility to adequately assess microstructural effects.

Radiation-Induced Chromium Depletion

In stainless steels, major alloying elements such as iron, nickel and chromium, are directly influenced by the flow of radiation-induced vacancies to grain boundaries.^{15,16} The slowest diffusing element, nickel, becomes enriched at sinks, while faster diffusers (chromium and iron) are depleted. Undersized minor elements and impurities such as silicon and phosphorus bind with interstitials and migrate preferentially to sinks.^{16,17} Grain boundary composition has been reported in a large number of neutron-irradiated stainless steels.^{7,18-25} At present, the most complete data has been generated using FEG-STEM with an incident electron probe diameter from 2 to 3 nm and through-foil thicknesses less than 75 nm. This results in an analysis resolution of about 3 to 5 nm that enables measurement of narrow, radiation-induced enrichment and depletion profiles.

Radiation-induced chromium depletion has been the focus of many IASCC studies because of its well documented effects in promoting IGSCC in sensitized stainless steels. Grain boundary chromium concentrations measured in neutron-irradiated 304, 316 and 348 SSs have been compiled and analyzed. Minimum chromium concentrations are plotted as a function of fluence in Figure 5. The data plotted has been adjusted to account for the effect of chromium-rich surface films on the measured concentrations. Grain boundary concentrations have been reduced by the difference (typically ~1%) between the measured matrix chromium and the bulk content. No attempt has been made to deconvolute the measured profiles to more accurately determine the true interfacial concentration for these narrow profiles. However, as noted above, the minimum chromium level measured has been used. Numerous reasons exist why FEG-STEM may underestimate RIS in neutron-irradiated stainless steels (particularly at low fluences), but there are few reasons for the opposite to be true. Therefore, the values plotted in Figure 5 are believed to better represent compositions at the boundaries which control SCC than an average of the two different boundaries typically examined. It is likely that the actual minimum concentrations are below those reported due to beam dilution effects.

The most detailed characterization of neutron fluence effects on grain boundary RIS has been reported by Jacobs¹⁸ on HP and CP 304 SSs as illustrated in Figure 5. A consistent decrease in interfacial chromium was detected with increasing fluence up to $\sim 2 \times 10^{21}$ n/cm², i.e. about 3 dpa. Samples irradiated to slightly higher fluences did not show a continued decrease in the grain boundary chromium level. This change in the rate of segregation is in agreement with the few high-fluence data points in Figure 5, with charged particle irradiations^{26,27} and with model predictions.^{14,28} Chromium enrichment has been reported at low fluence levels as indicated by the data of Asano, et al.¹⁹ This initial enrichment appears to shift the fluence dependence curve to higher chromium concentrations and require an increased fluence to achieve comparable grain boundary depletion. Considering the

wide range of materials and starting conditions, most data shows a consistent exponential decrease in chromium content with increasing fluence.

Conflicting information has been obtained for the segregation behavior of HP versus CP stainless steels. FEG-STEM measurements of Jacobs¹⁸ on 304 SS revealed no significant difference in chromium depletion. Kenik,²⁰ however, detected increased depletion in a CP versus a HP heat. Contrary to these measurements, Chung, et al.⁷ have reported much more chromium depletion in HP than in CP 304 SSs by Auger electron microscopy (AES). Grain boundary chromium contents were ~9 wt% in the HP heat and 14-16 wt% in the CP heats at fluence levels of 1.4 and 2×10^{21} n/cm² (~2 and 3 dpa), respectively. It is interesting that the AES results for the CP heats (14-16 wt%) fit well within the data trend in Figure 5, while the 9 wt% measured for the HP heats is much lower than other measurements. HP and CP 348 SSs have also been examined with the HP material showing similar depletion to the CP, but at about twice the fluence level.²¹

Chromium Depletion Effects on IGSCC

Intergranular SCC susceptibility of stainless steels depends on the grain boundary chromium concentration.²⁹⁻³¹ Bruemmer, et al.³⁰ documented a sharp change in behavior during SSR tests at a strain rate of 1×10^{-6} /s in 8 ppm O₂, 288°C water as the interfacial chromium content decreased from ~16 to 12 wt%. Intergranular fracture increased to >90% and the strain to failure dropped from 50 to 15% as shown in Figure 6. To make a direct comparison to SSR tests on irradiated stainless steels where strain rates have been performed at slower rates (1.6 to 3.7×10^{-7} /s), tests were conducted at a strain rate of 2×10^{-7} /s. All other test conditions were identical to those described previously.³⁰ The decrease in strain rate promoted nearly 100% IGSCC and strain to failure values of only 10% in specimens with chromium minimums less than 16 wt%. No IG cracking was observed in materials where grain boundaries revealed chromium levels similar to the matrix (~18%).

Data where radiation-induced chromium depletion and IGSCC have been measured are summarized in Figure 7. The SSR tests are limited, but indicate that some level of depletion exists in all stainless steels which fail by IGSCC. As the grain boundary chromium concentration drops below ~16 wt%, 304 SS becomes susceptible to cracking under the specific conditions of the test. The few data points for 316 SS suggest a lower minimum, corresponding to a interfacial chromium depletion of ~2 wt% below the matrix, consistent with the 304 SS results. Included in Figure 7 is Jacobs¹⁸ in-core eddy current measurements to indicate the extent of IGSCC based on the percentage of cracked components. These measurements on a single HP 304 SS heat revealed initial cracking at a fluence of $\sim 1 \times 10^{21}$ n/cm² with most components showing cracks by 3×10^{21} n/cm². Although many differences are present including dynamic irradiation, loading mode and creviced conditions, results for the in-core tests are in good agreement with the depletion-cracking comparisons from SSR tests.

The relationship established between chromium depletion and IGSCC from Figure 6 has been plotted with the irradiated materials data in Figure 7. All irradiated specimens that show IG cracking have sufficient grain boundary chromium depletion for IGSCC susceptibility in the unirradiated condition. Thus, chromium depletion can explain the observations of IGSCC without the necessity to consider other radiation effects on microstructure and microchemistry. Differences noted in Figures 2 and 3 for the IGSCC versus fluence data of Chung, Jacobs and Kodama can be explained based on their measurements of chromium depletion. In addition, the

variations among several heats in the yield strength correlation agree with measured interfacial chromium levels. For example, the high IGSCC-susceptibility HP heat of Chung corresponds to lowest measured chromium content (9%), while the IGSCC-resistant CP material (sheath) has only slight depletion (16%). The reduced degree of IGSCC in Chung's heats are probably due to the less aggressive test environment. Kodama's IGSCC results are also in excellent agreement with measured chromium depletion. Grain boundary enrichment of chromium before irradiation delays depletion development (and IGSCC) to higher fluences.

However, questions still remain as to why a few chromium-depleted stainless steels show more resistance to IGSCC. Jacobs, et al.¹⁰ have reported that grain boundary chromium measurements are not consistent with SSR tests of high-temperature solution-annealed (HTSA) 304 SS irradiated to fluences of $\sim 3 \times 10^{21}$ n/cm² (4 dpa). Three irradiated specimens were found to have similar minimum chromium contents (~ 15 wt%), but only one exhibited significant IGSCC. This depletion level appears to be near the transition in cracking response. The HTSA treatments (1204-1316°C) produced very large grain sizes (up to 300 μ m) which can have a direct effect on crack initiation and propagation. Depleted specimens resistant to cracking also had much higher RIS of silicon. Segregation of silicon or phosphorus has been shown to improve IGSCC resistance in proton-irradiated 304 SS.^{32,33} This potential beneficial effect of segregated silicon is opposite to its proposed effect on the IGSCC susceptibility of CP 348 SS.²¹

In summary, radiation-induced chromium depletion appears to play a primary role in the IASCC susceptibility of austenitic stainless steels. Grain boundary depletion below a critical chromium concentration of about 16 wt% is required for cracking. Other microstructural and microchemical variables will influence the relationship between chromium depletion and IGSCC. Although direct comparisons between grain boundary composition and IASCC have increased dramatically, much more data is required to establish specific cracking relationships. Tests must be performed on well-characterized materials with systematic variations in bulk composition.

Conclusions

Observed differences in the neutron fluence dependence of IGSCC for various stainless steel heats were examined by comparisons to yield strength and/or grain boundary chromium concentration. Cracking of certain heats at lower fluences was consistent with higher yield strengths. Most BWR-irradiated stainless steels showed a critical yield strength for susceptibility of ≥ 600 MPa, but others (primarily ATR-irradiated) experienced cracking at lower yield strengths. Critical yield strengths for cracking ranged from 450 to 800 MPa, representing strength increases from 2 to 4 times the annealed value. Although the response of individual materials was improved, a general correlation between radiation hardening and IGSCC susceptibility was not found for all heats.

Comparisons between measured chromium contents and SCC revealed that materials showing IG cracking have significant ($\geq 2\%$) depletion. Depletion levels to cause cracking (below ~ 16 wt%) are in good agreement with similar SCC tests on unirradiated 304 SS with controlled grain boundary chromium concentrations. Heats that prompt variability in the yield strength correlation, are accounted for by differences in their interfacial chromium contents. Certain stainless steels are more resistant to cracking even though they have significant radiation-induced chromium depletion. It is proposed that chromium depletion is required for IASCC, but observed susceptibility is modified by other microchemical and microstructural components.

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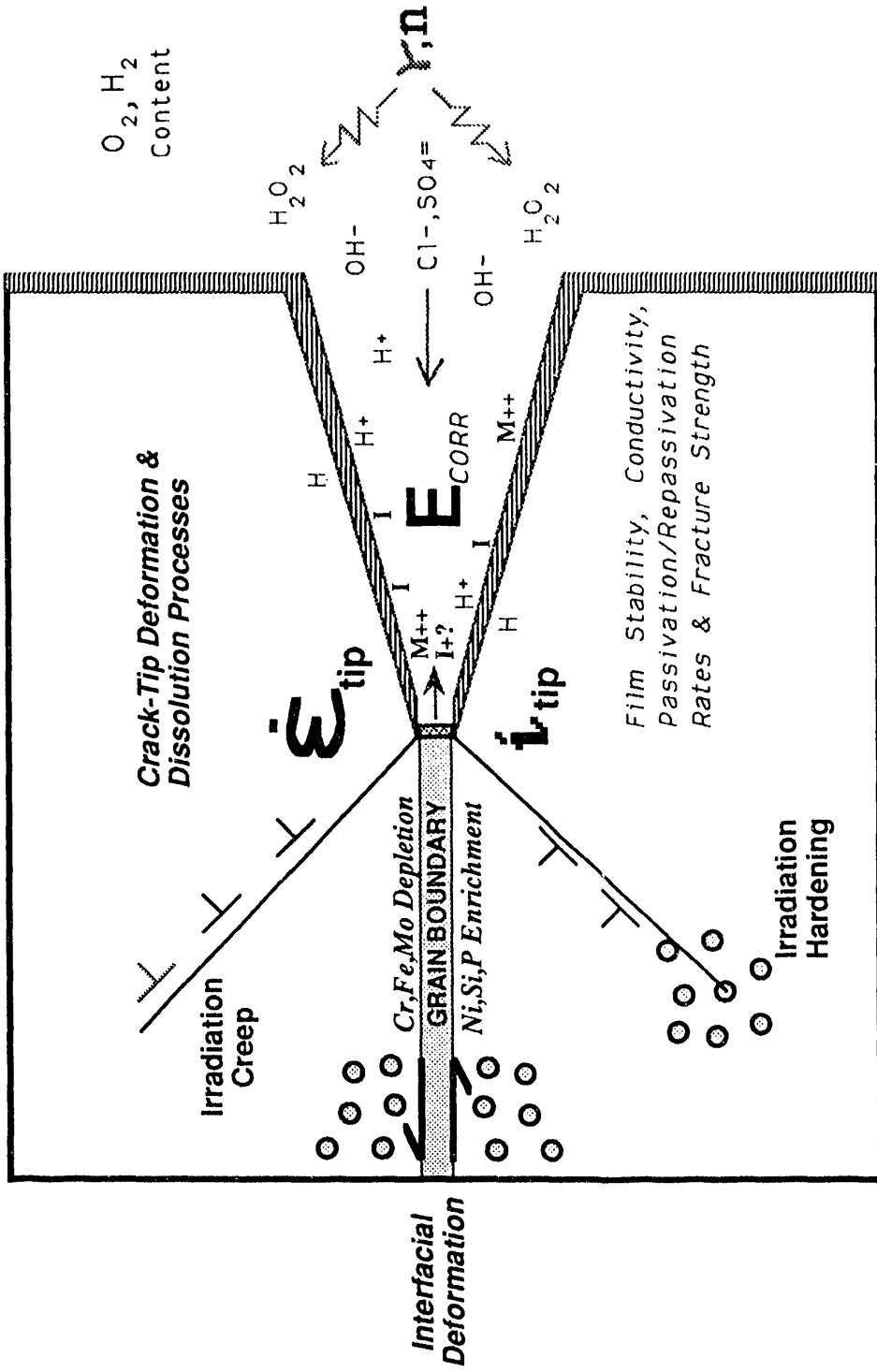


Figure 1. Schematic Illustrating Possible Radiation Effects on the IGSCC Susceptibility of an Austenitic Stainless Steel in High-Temperature Water Environments.

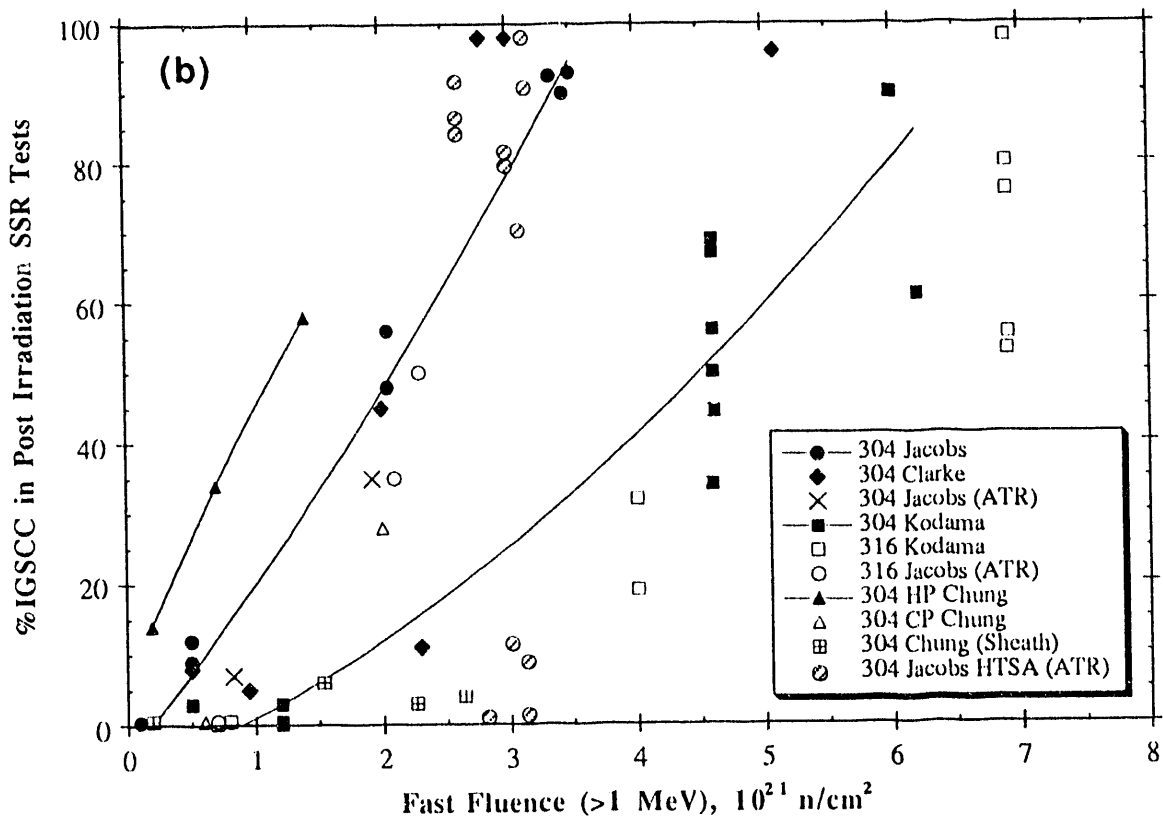
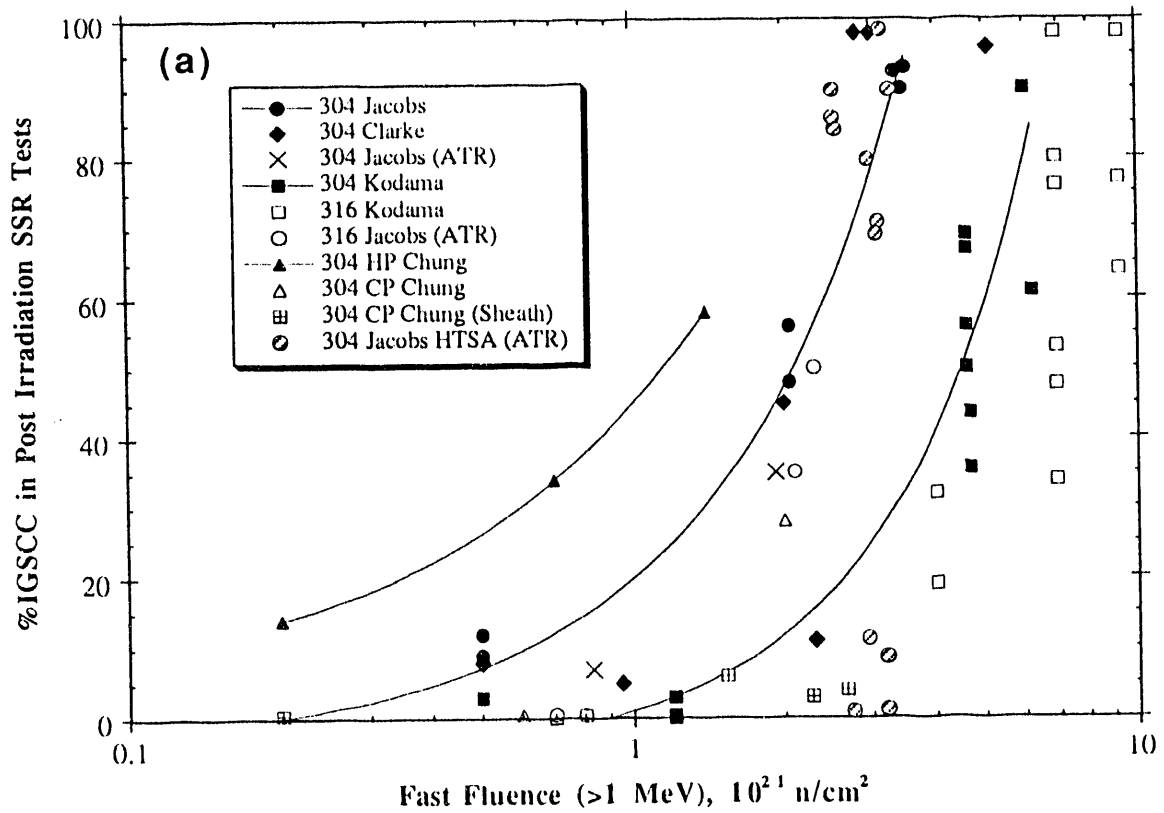


Figure 2. IGSCC Susceptibility of Irradiated Stainless Steels as a Function of Fast Neutron Fluence on Log (a) and Linear (b) Scale.

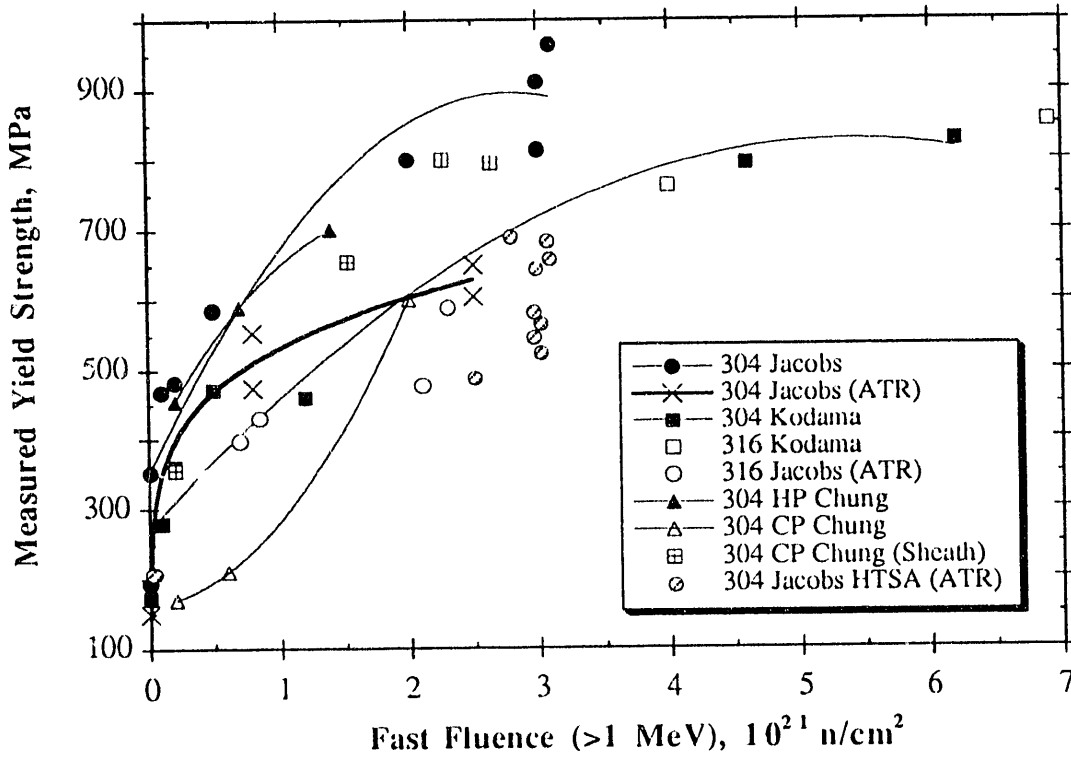


Figure 3. Fast Neutron Fluence Effects on Yield Strength for 304 and 316 SSs.

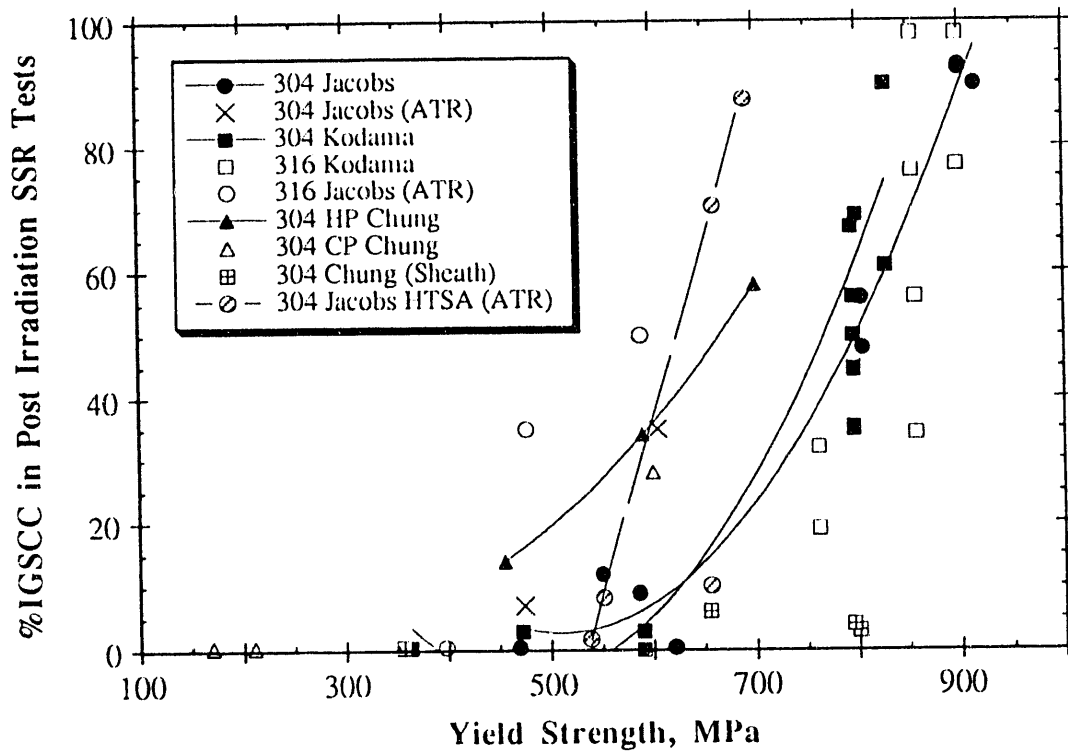


Figure 4. Correlation Between Yield Strength and IGSCC for Neutron-Irradiated Stainless Steels.

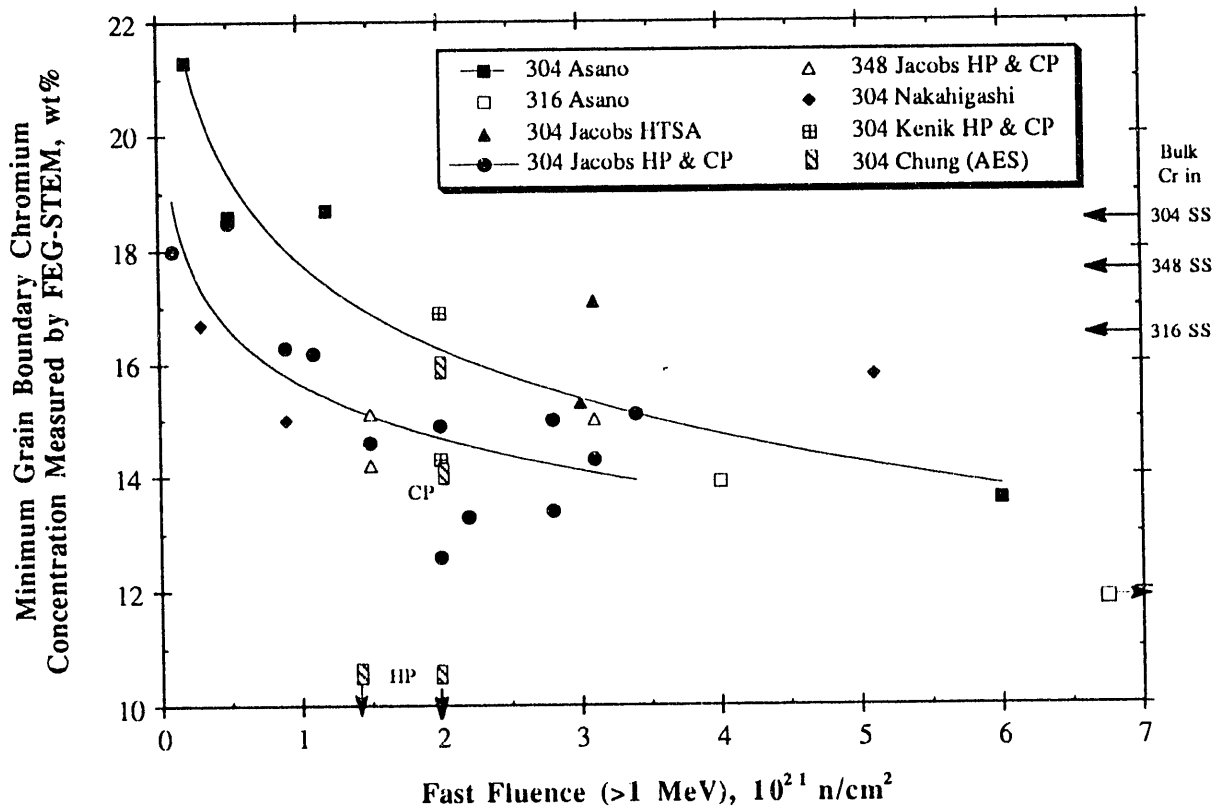


Figure 5. Measured Grain Boundary Chromium Concentration as a Function of Fluence.

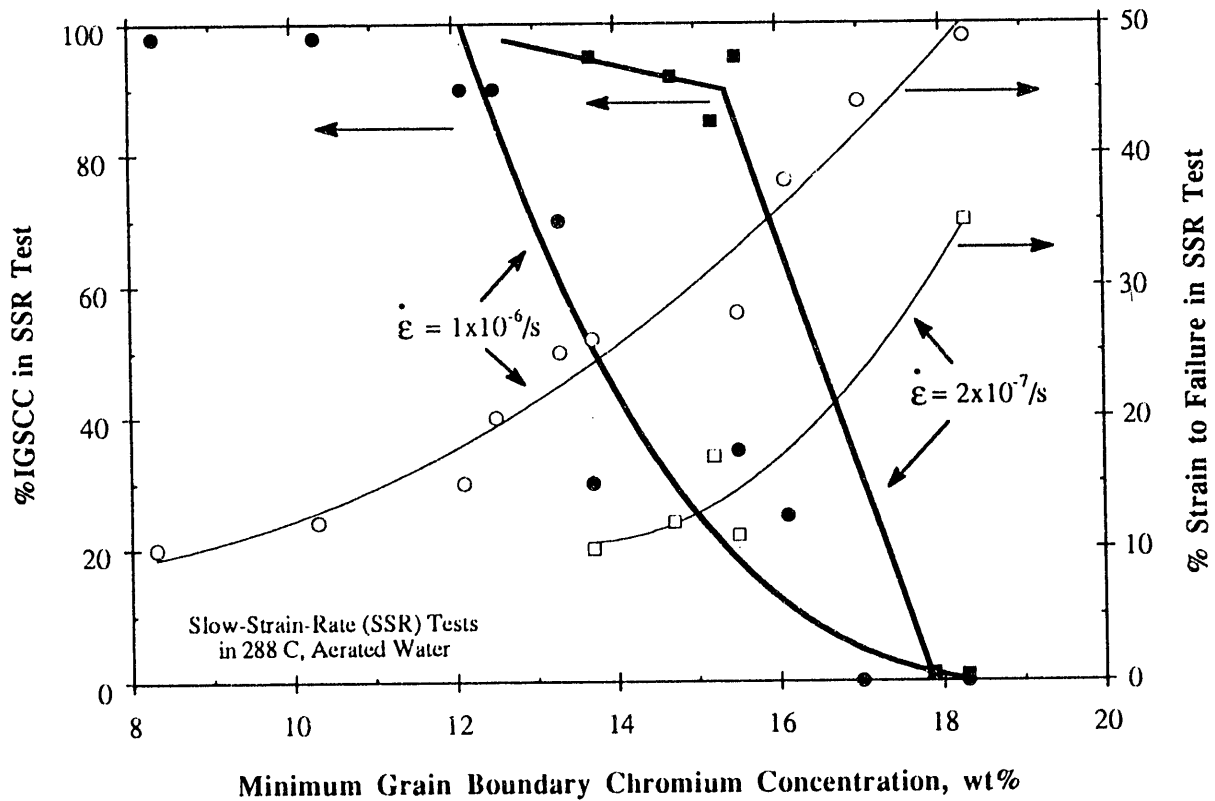


Figure 6. Influence of Grain Boundary Chromium Concentration on IGSCC Susceptibility of Thermally Treated 304 SS at Two Different Strain Rates.

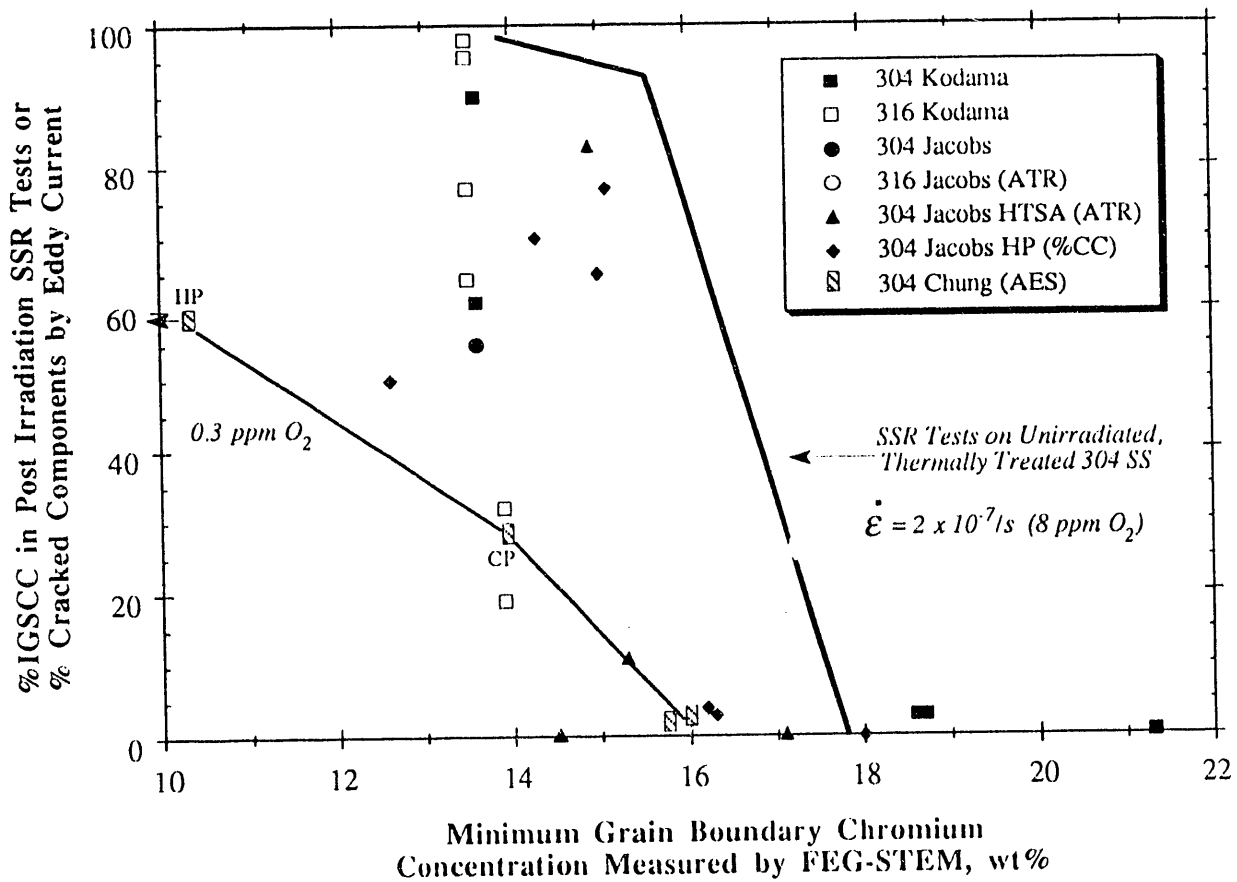


Figure 7. Comparison Between IGSCC Susceptibility and Measured Grain Boundary Chromium Concentration.

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