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Heavy-Section Steel Irradiation Program

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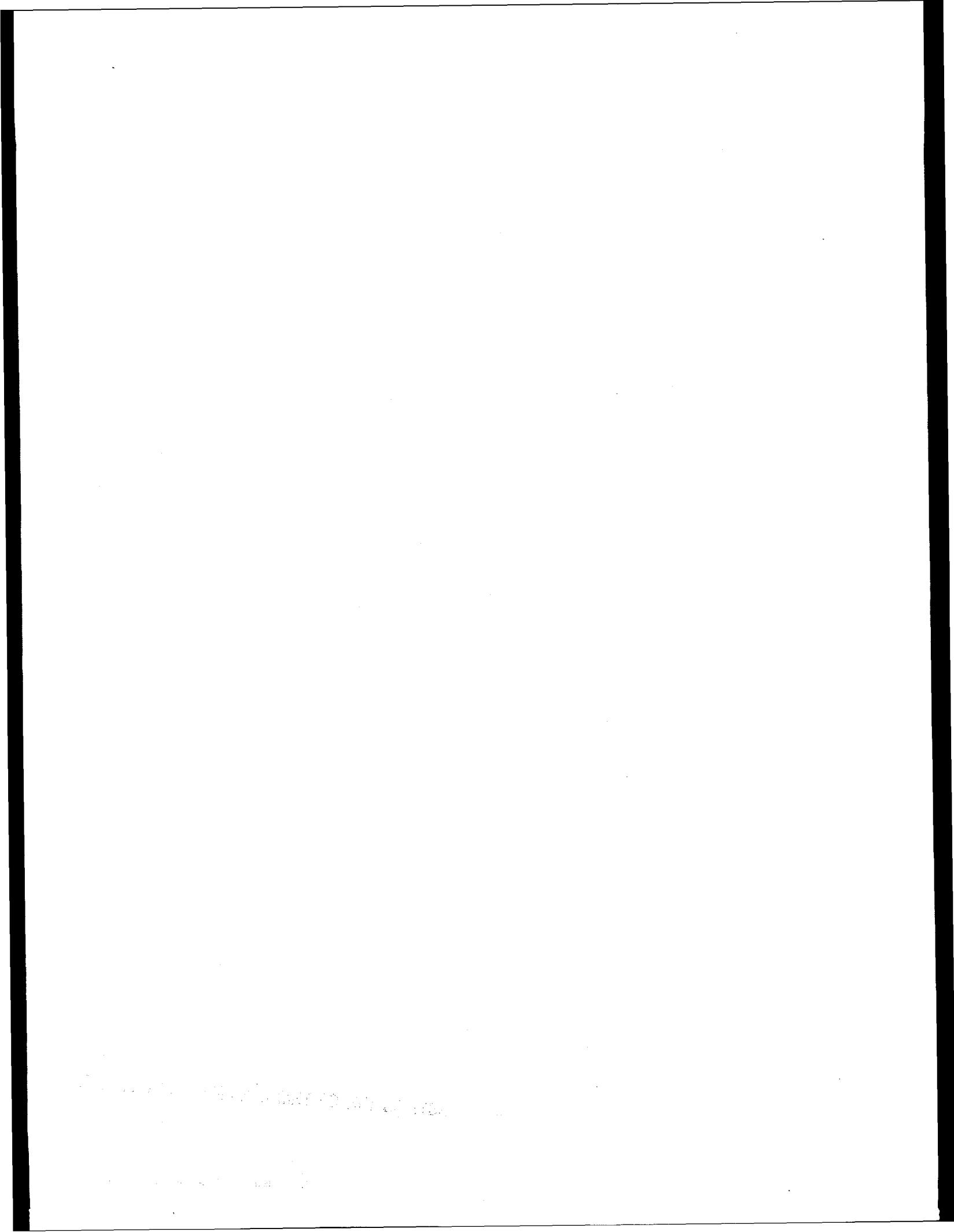
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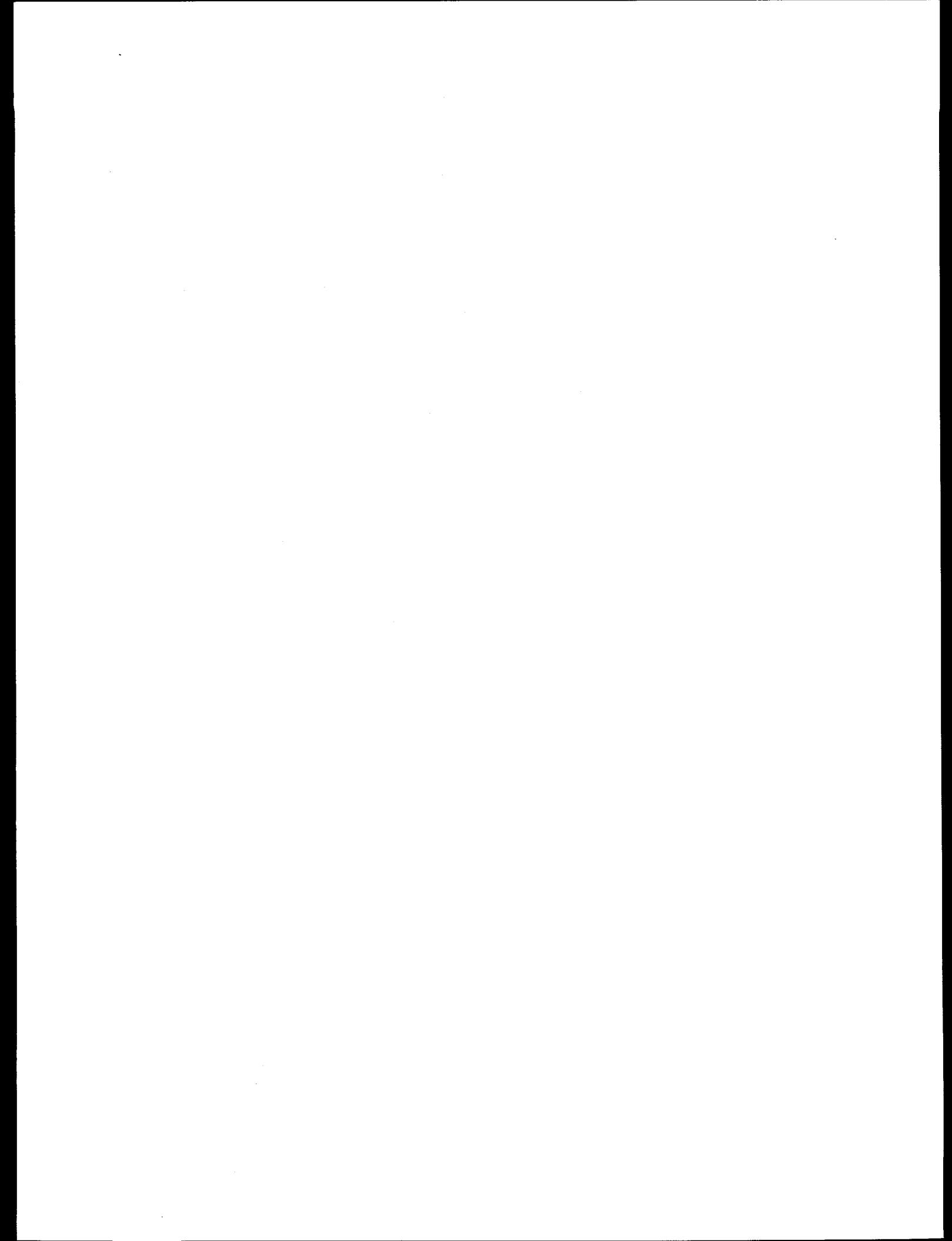
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Abstract

Maintaining the integrity of the reactor pressure vessel (RPV) in a light-water-cooled nuclear power plant is crucial in preventing and controlling severe accidents which have the potential for major contamination release. The RPV is the only key safety-related component of the plant for which a duplicate or redundant backup system does not exist. It is therefore imperative to understand and be able to predict the capabilities and limitations of the integrity inherent in the RPV. In particular, it is vital to fully understand the degree of irradiation-induced degradation of the RPV's fracture resistance which occurs during service, since without that radiation damage, it is virtually impossible to postulate a realistic scenario that would result in RPV failure.

For this reason, the Heavy-Section Steel Irradiation (HSSI) Program has been established with its primary goal to provide a thorough, quantitative assessment of the effects of neutron irradiation on the material behavior and, in particular, the fracture toughness properties of typical pressure-vessel steels as they relate to light-water reactor pressure-vessel integrity. The program includes the direct continuation of irradiation studies previously conducted within the Heavy-Section Steel Technology Program augmented by enhanced examinations of the accompanying microstructural changes. Effects of specimen size; material chemistry; product form and microstructure; irradiation fluence, flux, temperature, and spectrum; and postirradiation annealing are being examined on a wide range of fracture properties. The HSSI Program is arranged into 14 tasks: (1) program management, (2) fracture toughness (K_{Ic}) curve shift in high-copper welds, (3) crack-arrest toughness (K_{Ia}) curve shift in high-copper welds, (4) irradiation effects on cladding, (5) K_{Ic} and K_{Ia} curve shifts in low upper-shelf (LUS) welds, (6) annealing effects in LUS welds, (7) irradiation effects in a commercial LUS weld, (8) microstructural analysis of irradiation effects, (9) in-service aged material evaluations, (10) correlation monitor materials, (11) special technical assistance, (12) Japan Power Development Reactor steel examination, (13) technical assistance for Joint Coordinating Committee on Civilian Nuclear Reactor Safety (JCCCNRS) Working Groups 3 and 12, and (14) additional requirements for materials.

During this period, the duplex-type crack-arrest specimen tests from Phase II of the K_{Ia} program were evaluated and a report on their test results prepared for publication. A new, remote crack-arrest fixture for testing the large, irradiated specimens from the Italian Committee for Research and Development of Nuclear Energy and Alternative Energies (ENEA) was built. To determine if the previously determined increase in the annealed upper-shelf energy of weld 73W CVN specimens is due to thermal aging, unirradiated specimens of HSSI 73W and Russian VVER-440 welds were aged in air at 460 and 490°C (860 and 915°F) for 168 h, and it appears that the aging accounts for most of the increase. Crack-arrest tests were made on the Midland beltline WF-70 weld metal. A draft NUREG report, *Unirradiated Material Properties of Midland Weld WF-70*, was prepared, describing that while Charpy V and drop-weight nil-ductility transition temperatures indicated that the nozzle course and beltline WF-70 weld metals had similar fracture toughness, the fracture mechanics tests and tensile properties indicated a significant difference. Irradiation of the low-fluence, scoping capsules of the Midland weld was completed and irradiation of the second large capsule (10.06) begun. The Charpy and tensile specimens exposed to low fluence levels in the cavity outside the reactor vessel at the Trojan Reactor were tested, and their Charpy V-notch (CVN) impact energy values were indistinguishable from those of unirradiated material. Model alloys were acquired and their preliminary transmission electron microscopy (TEM) examination performed to examine microstructure/mechanical property correlation following ion irradiation to determine the dislocation barrier strength of small point-defect clusters in irradiated steels. An extensive set of molecular dynamics calculations were carried out to investigate the evolution of displacement cascades in iron. Bids were reviewed and a purchase order issued for the acquisition of a computer numerically controlled machining center suitable for hot-cell operations. A literature review and analysis of subscale Charpy impact specimen designs, procedures, and data was conducted and a test matrix developed to study the effects of different geometrical parameters on the relationship between subscale and full-size specimens. Oak Ridge National Laboratory participated in and coordinated the initial activities of a JCCCNRS Working Group 3 round-robin program on J-R testing. CVN impact and tensile testing was performed on stainless steel welds with up to 20,000-h thermal exposure at 343°C (650°F). Optical metallography, scanning electron microscopy, TEM, and atom-probe/field-ion microscopy were used to follow changes in their microstructures as a consequence of thermal exposure.



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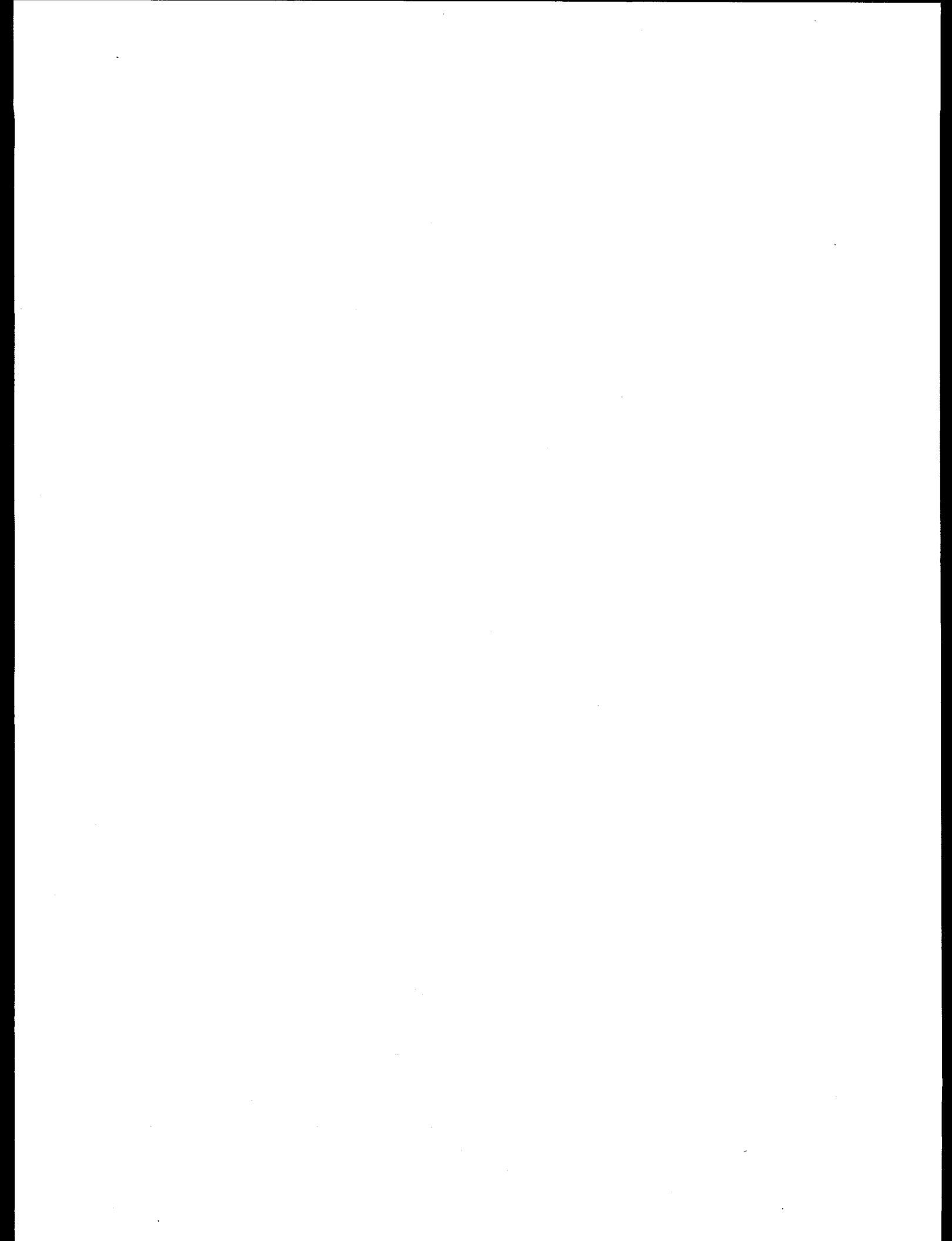
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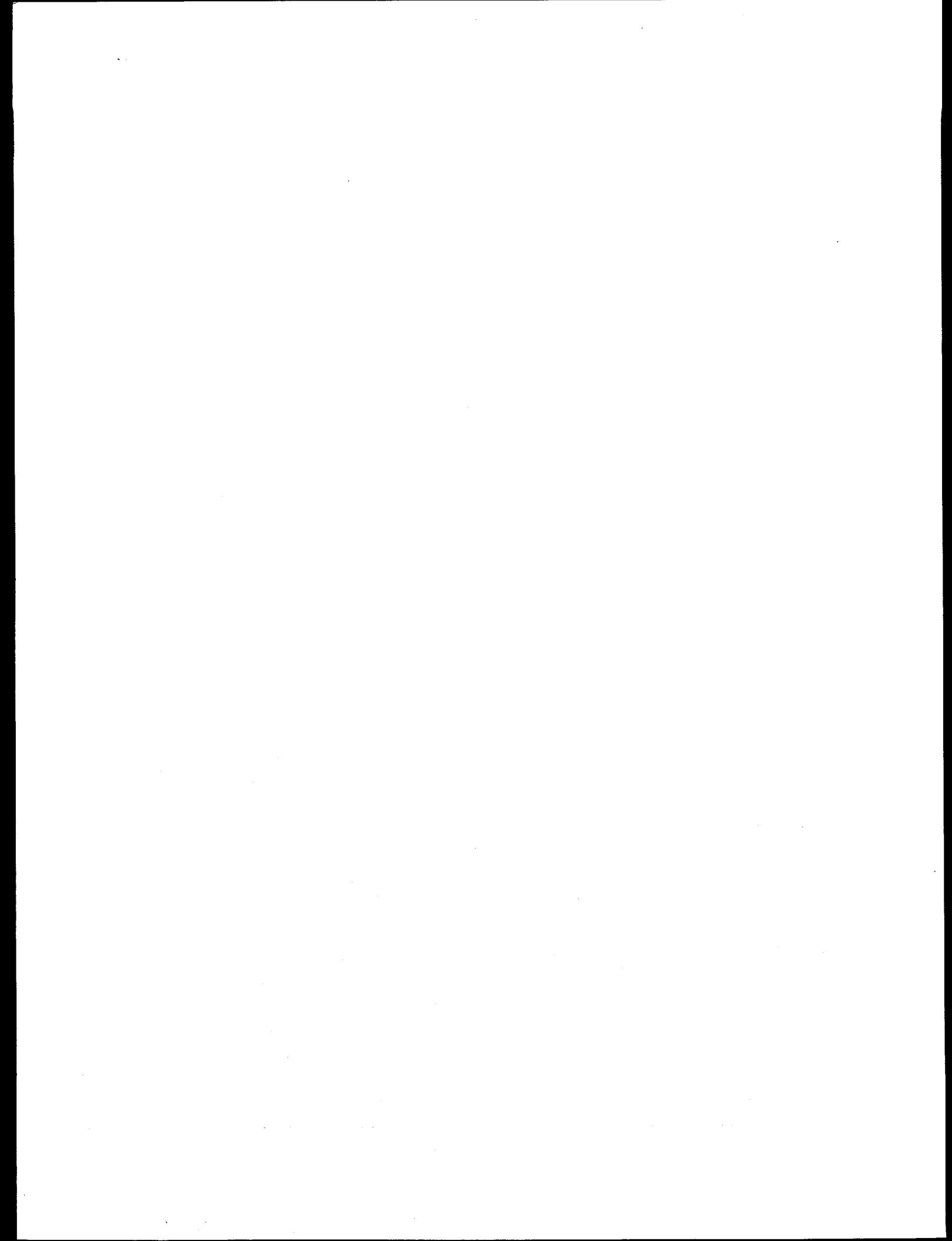
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Preface

The primary goal of the Heavy-Section Steel Irradiation (HSSI) Program is to provide a thorough, quantitative assessment of the effects of neutron irradiation on the material behavior and, in particular, the fracture toughness properties of typical pressure-vessel steels as they relate to light-water reactor pressure vessel (RPV) integrity. The program includes studies of the effects of irradiation on the degradation of mechanical and fracture properties of vessel materials augmented by enhanced examinations and modeling of the accompanying microstructural changes. Effects of specimen size; material chemistry; product form and microstructure; irradiation fluence, flux, temperature, and spectrum; and postirradiation annealing are being examined on a wide range of fracture properties. Results from the HSSI studies will be incorporated into codes and standards directly applicable to resolving major regulatory issues which involve RPV irradiation embrittlement such as pressurized-thermal shock, operating pressure-temperature limits, low-temperature overpressurization, and the specialized problems associated with low upper-shelf welds.

This HSSI Program progress report covers work performed from April through September 1993. The work performed by Oak Ridge National Laboratory (ORNL) is managed by the Metals and Ceramics (M&C) Division of ORNL. Major tasks at ORNL are carried out by the M&C, Computing Applications, and Engineering Technology Divisions.

Previous HSSI Progress Reports in this series are:

- NUREG/CR-5591, Vol. 1, No. 1
(ORNL/TM-11568/V1&N1)
- NUREG/CR-5591, Vol. 1, No. 2
(ORNL/TM-11568/V1&N2)
- NUREG/CR-5591, Vol. 2, No. 1
(ORNL/TM-11568/V2&N1)
- NUREG/CR-5591, Vol. 2, No. 2
(ORNL/TM-11568/V2&N2)
- NUREG/CR-5591, Vol. 3
(ORNL/TM-11568/V3)
- NUREG/CR-5591, Vol. 4, No. 1
(ORNL/TM-11568/V4&N1)

Some of the series of irradiation studies conducted within the HSSI Program were begun under the Heavy-Section Steel Technology (HSST) Program prior to the separation of the two programs in 1989. Previous HSST Program progress reports contain much information on the irradiation assessments being continued by the HSSI Program as well as earlier related studies. The HSST Program progress reports issued before formation of the HSSI Program are also tabulated here as a convenience to the reader.

- ORNL-4176
- ORNL-4315
- ORNL-4377
- ORNL-4463
- ORNL-4512
- ORNL-4590
- ORNL-4653
- ORNL-4681
- ORNL-4764
- ORNL-4816
- ORNL-4855
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- ORNL-4971
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Summary

1. Program Management

The Heavy-Section Steel Irradiation (HSSI) Program is arranged into 14 tasks: (1) program management, (2) fracture toughness (K_{Ic}) curve shift in high-copper welds, (3) crack-arrest toughness (K_{Ia}) curve shift in high-copper welds, (4) irradiation effects on cladding, (5) K_{Ic} and K_{Ia} curve shifts in low upper-shelf (LUS) welds, (6) annealing effects in LUS welds, (7) irradiation effects in a commercial LUS weld, (8) microstructural analysis of irradiation effects, (9) in-service aged material evaluations, (10) correlation monitor materials, (11) special technical assistance, (12) Japan Power Development Reactor (JPDR) steel examination, (13) technical assistance for Joint Coordinating Committee on Civilian Nuclear Reactor Safety (JCCNRS) Working Groups 3 and 12, and (14) additional requirements for materials. Report chapters correspond to the tasks. The work is performed by the Oak Ridge National Laboratory (ORNL). During the report period, 18 technical presentations were given, 6 technical papers were published, and 3 foreign trip reports were issued.

2. K_{Ic} Curve Shift in High-Copper Welds

The objectives of the Fifth Irradiation Series are to determine the K_{Ic} curve shifts and shapes for two irradiated high-copper submerged-arc welds (SAWs). Phase I, with a large number of specimens irradiated to $\sim 1.5 \times 10^{19}$ neutrons/cm² (>1 MeV), has been completed and reported. The objective of Phase II is to obtain results at a fluence of 5×10^{19} neutrons/cm² for evaluation of curve shift and shape at high fluence. A specimen matrix has been developed, archive materials have been identified, and fabrication of specimens will be initiated following completion of the irradiation capsule design.

3. K_{Ia} Curve Shift in High-Copper Welds

The objectives of the Sixth Irradiation Series are to determine the K_{Ia} curve shifts and shapes for two high-copper SAWs. The program was conducted in two phases. In Phase I, 36 weld-embrittled-type crack-arrest specimens were tested, and detailed results with some preliminary conclusions have been published. In Phase II of the K_{Ia} program, 24 duplex-type crack-arrest specimens were tested. Charpy V-notch (CVN) specimens irradiated in the same capsules as the crack-arrest specimens were also tested, and a 41-J transition temperature shift was determined from these specimens. Two "mean" curves of the same form as the American Society of Mechanical Engineers (ASME) K_{Ia} curve were fit to the unirradiated and irradiated data with only a reference temperature as a parameter. The shift between the two curves agrees well with the 41-J transition temperature shift obtained from the CVN specimen tests. Moreover, the four data points resulting from tests on the duplex crack-arrest specimens of the present study did not make a significant change to the curve fits made to either the previously obtained weld-embrittled specimen K_a data or all the weld-embrittled and duplex K_a data combined. A report on the test results of Phase II is in the final stages of preparation for publication. A new, remote crack-arrest fixture for testing the large, irradiated specimens from the Italian Committee for Research and Development of Nuclear Energy and Alternative Energies (ENEA) has been built and has undergone some evaluation.

4. Irradiation Effects in Cladding

The objective of this series is to obtain toughness properties of stainless steel cladding in the unirradiated and irradiated conditions. The properties obtained include tensile, CVN impact, and J-integral toughness. The goal is to evaluate the fracture resistance of irradiated weld-metal cladding representative of that used in early pressurized-water reactors. The fracture properties are needed for detailed integrity analyses of vessels during overcooling situations. There was no significant activity within this task during this reporting period.

5. K_{Ic} and K_{Ia} Curve Shifts in LUS Welds

The primary objective of Series 8 is to examine the K_{Ic} and K_{Ia} for LUS high-copper weld metal irradiated at 288°C, with particular emphasis on the shift and change shape of the ASME curves following irradiation. During the current reporting period, the assessment of the welding consumables to be used in the fabrication of the high-copper, LUS weld to be examined was continued, and plans were made to produce a test weld to ascertain its experimental suitability. In general, the LUS welds tend to give RT_{NDTs} that are controlled by the CVN behavior. Use of the existing inventory of weld wire that was used for fabrication of HSSI weld 73W is one consideration. Use of Linde 80 welding flux with that wire would likely produce a high-copper (~0.31%) weld with LUS energy but would likely deplete the wire inventory. Other options are also being pursued, and a trial weld will be fabricated during 1994.

6. Annealing Effects in Low Upper-Shelf Welds

The purpose of the Ninth Irradiation Series is to evaluate the correlation between fracture toughness and CVN impact energy during irradiation, annealing and reirradiation (IAR). Results from previously reported tests on irradiated and annealed HSSI weld 73W CVN specimens have exhibited an approximate 25% increase in upper-shelf energy (USE) compared to that in the unirradiated condition. In order to determine if this increase is due to thermal aging, unirradiated specimens of HSSI 73W and Russian VVER-440 welds were aged in air at 460 and 490°C (860 and 915°F) for 168 h. It appears that aging may account for most of the increase exhibited by the irradiated and annealed specimens. It also appears that aging at the temperatures and times mentioned above did have a significant effect on the impact energy in the upper transition regions of the VVER-440 weld, which contained 0.030% P, but not on the HSSI 73W, which had only 0.005% P. Both welds had received a postweld heat treatment (PWHT), the HSSI 73W at 607°C (1125°F) for at least 40 h and the VVER-440 at 670°C (1240°F) for 34 h. Thus, it seems unlikely that relatively short-term aging at a lower temperature would significantly alter the microstructure. Plans are to examine the unirradiated material in both the as-fabricated and annealed conditions to determine whether a microstructural basis for the increase in the USE exists.

7. Irradiation Effects in a Commercial LUS Weld

The primary objective of Series 10 is to investigate the postirradiation fracture toughness of the LUS, high-copper SAW from the beltline region of the Midland Unit 1 reactor vessel. The weld from that vessel is of considerable interest because it carries the Babcock and Wilcox designation WF-70, an SAW fabricated with a specific heat of weld wire and specific lot of flux. Welds with the WF-70 designation are the controlling material (regarding irradiation effects) in several operating nuclear plants. During the current reporting period, crack-arrest tests were made on the Midland beltline WF-70 weld metal. Because the crack-starter weld bead produced heat-affected zone (HAZ) material of improved toughness, most running cracks were initiated only after some prior slow-stable crack growth through the HAZ. Consequently, most post-crack-arrest remaining ligaments were too small to yield valid K_{Ia} , according to American Society for Testing and Materials (ASTM) E 1221-88. Nevertheless, it is believed that the results produced some viable values. This effort is continuing using duplex crack-arrest specimens.

A draft NUREG report, *Unirradiated Material Properties of Midland Weld WF-70*, has been prepared. Although the widely used empirical test methods to establish transition temperature (Charpy V and drop-weight nil-ductility transition) indicated that the nozzle course and beltline WF-70 weld metals had identical fracture toughness; the fracture mechanics tests and tensile properties indicated a significant difference. The nozzle course weld had a 27°C (49°F) higher transition temperature. J-R curves were developed and compared to a Charpy correlation model developed by Eason et al. In general, the Charpy-based prediction of the experimental J-R curves worked quite well.

Scoping capsules were constructed for the WF-70 nozzle and beltline welds for irradiation to 0.5×10^{19} neutrons/cm² (>1 MeV). Irradiation of the scoping capsules and the first large capsule, 10.05, was completed. Large specimen capsule 10.06, 2T size, has been assembled, and the irradiation to 1×10^{19} neutrons/cm² (>1 MeV) is now in progress.

8. Microstructural Analysis of Irradiation Effects

The overall long-term goal of this task is to develop a physically based model which can be used to predict irradiation-induced embrittlement in reactor vessel steels over the full range of their service conditions. The model should be tethered soundly on the microstructural level by results from advanced microstructural analysis techniques and constrained at the macroscopic level to produce predictions consistent with the large array of macroscopic embrittlement measurements that are available. During this reporting period, the Charpy and tensile specimens that had been exposed in the cavity outside the reactor vessel at the Trojan Reactor were tested. The CVN impact energy values at the low fluence attained are indistinguishable from that of unirradiated material. Nine model alloys were acquired for use in a study of microstructure/mechanical property correlation following ion irradiation to determine the dislocation barrier strength of small point defect clusters in irradiated steels. These clusters can be responsible for much of the hardening observed in these materials. The preliminary transmission electron microscopy (TEM) examination of the alloys in the as-received condition and after the first ion irradiations were completed. An extensive set of molecular dynamics (MD) calculations has been carried out to investigate the evolution of displacement cascades in iron. The results of the MD simulations were used to refine cascade survival and clustering parameters in the point defect clustering model that is being developed under this task.

9. In-Service Aged Material Evaluations

The overall objective of this task is to assess the service-induced degradation of fracture resistance through examination of components exposed during in-nuclear-plant operation. The initial focus of this task is to augment the existing hot-cell testing capability available to the HSSI Program with remote machining capabilities for the fabrication of specimens from samples of activated steel obtained from service-exposed components. During this reporting period, the acquisition of computer numerically controlled (CNC) machining centers suitable for hot-cell operations was pursued. In response to our specification, one bid was received from Emco Maier, Inc. (Columbus, Ohio), for a model VMC-100 machine. After appropriate technical and administrative review, an order was placed for purchase of this machine. The contract includes a demonstration machining of specific specimen geometries.

10. Correlation Monitor Materials

This is a task that has been established with the explicit purpose of ensuring the continued availability of the pedigreed and extremely well-characterized material now required for inclusion in all additional and future surveillance capsules in commercial light-water reactors. Having recognized that the only remaining materials qualified for use as a correlation monitor in reactor surveillance capsules are the pieces remaining from the early Heavy-Section Steel Technology plates 01, 02, and 03, this task will provide for cataloging, archiving, and distributing the material on behalf of the Nuclear Regulatory Commission (NRC). During this reporting period, the task of moving the material, previously identified and inventoried as correlation monitor material during the previous reporting period, from its current site at the Y-12 Plant to a controlled-access storage location at ORNL was initiated.

11. Special Technical Assistance

This task has been included with the HSSI Program to provide a vehicle in which to conduct and monitor short-term, high-priority subtasks. During the current reporting period, the HSSI Program has been participating in an ASTM E-10 (Subcommittee E10.02) project on reconstitution of CVN specimens. The ORNL contribution is to

perform all specimen testing but does not include reconstituting the specimens. All the baseline tests were completed with both the ASTM and International Organization for Standardization strikers. Broken specimens were distributed to the participating laboratories by the ASTM task leader, and testing will be performed when at least six participants have supplied specimens.

12. Evaluation of Steel from the JPDR Pressure Vessel

There is a need to validate the results of irradiation effects research by the examination of material taken directly from the wall of a pressure vessel which has been irradiated during normal service. This task has been included with the HSSI Program to provide just such an evaluation on material from the wall of the pressure vessel from the JPDR. During this reporting period, very slow movement toward the production of a signed, formal agreement was made. ORNL was given assurances by the Japan Atomic Energy Research Institute that this is typical for agreements with them and does not indicate any lack of interest or other show-stopping problems from their point of view. It is anticipated that the agreement should be finalized during the next reporting period.

In anticipation of the implementation of the JPDR, which will include the need to test subsize impact specimens, efforts were initiated for the fabrication and testing of the subsize specimens. A literature review and analysis of subsize Charpy impact specimen designs, procedures, and data was conducted. A test matrix was developed and materials identified to study the effects of different geometric parameters on the relationship between subsize and full-size specimens.

13. Technical Assistance for JCCCNRS Working Groups 3 and 12

The purpose of this task is to provide technical support for the efforts of the U.S.-Russian JCCCNRS Working Group 3 on radiation embrittlement and Working Group 12 on aging. Specific activities under this task are: (1) supply of materials and preparation of test specimens for collaborative IAR studies to be conducted in Russia; (2) capsule preparation and initiation of irradiation of Russian specimens within the United States; (3) preparation for, and participation in, Working Groups 3 and 12 meetings; and (4) sponsoring of the assignment of M. A. Sokolov of the Russian National Research Center-Kurchatov Institute.

Regarding irradiation experiments, specimens of two materials supplied by ORNL are under irradiation in the Novovoronezh Unit 5 reactor. Additionally, specimens supplied by Russia were encapsulated in HSSI capsule 10.06 and are being irradiated in the University of Michigan Ford Nuclear Reactor. ORNL is participating in and coordinating a Working Group 3 round-robin program on J-R testing. Specimen blanks have been machined and sent to the Kurchatov Institute.

14. Additional Requirements for Materials

The purpose of this task is to provide technical expertise and assistance in the review of national codes and standards that may be referenced in NRC regulations or guides related to nuclear reactor components. The specific activities to be performed include (1) review of new materials and requirements proposed for inclusion into national codes and standards, of ASME code cases, and of potential deficiencies in proposed supporting technology and data; (2) continuation of long-term aging of stainless steel welds; and (3) continuation of the low-temperature PWHT study of low-alloy steels. The primary activity on this task has involved the thermal aging of stainless steel weld metal. CVN impact and tensile testing has been performed on welds with up to 20,000-h exposure at 343°C (650°F). Additionally, optical metallography, scanning electron microscopy, TEM, and atom-probe field-ion microscopy have been used to follow changes in the microstructures as a consequence of exposure.

Heavy-Section Steel Irradiation Program Semiannual Progress Report for April through September 1993[†]

W. R. Corwin

1. Program Management

The Heavy-Section Steel Irradiation (HSSI) Program, a major safety program sponsored by the Nuclear Regulatory Commission (NRC) at Oak Ridge National Laboratory (ORNL), is an engineering research activity devoted to providing a thorough, quantitative assessment of the effects of neutron irradiation on the material behavior, particularly the fracture toughness properties, of typical pressure-vessel steels as they relate to light-water reactor (LWR) pressure-vessel integrity. The program centers on experimental assessments of irradiation-induced embrittlement [including the completion of certain irradiation studies previously conducted by the Heavy-Section Steel Technology (HSST) Program] augmented by detailed examinations and modeling of the accompanying microstructural changes. Effects of specimen size; material chemistry; product form and microstructure; irradiation fluence, flux, temperature, and spectrum; and postirradiation annealing are being examined on a wide range of fracture properties. Fracture toughness (K_{Ic} and J_{Ic}), crack-arrest toughness (K_{Ia}), ductile tearing resistance (dJ/da), Charpy V-notch (CVN) impact energy, drop-weight (DWT) nil-ductility transition (NDT), and tensile properties are included. Models based on observations of radiation-induced microstructural changes using the atom probe field-ion microscope and the high-resolution transmission electron microscope (TEM) are being developed to provide a firm basis for extrapolating the measured changes in fracture properties to wide ranges of irradiation conditions. The principal materials examined within the HSSI Program are high-copper welds because their postirradiation properties frequently limit the continued safe operation of commercial reactor pressure vessels (RPVs). In addition, a limited effort will focus on stainless steel weld-overlay cladding typical of that used on the inner surfaces of RPVs because its postirradiation fracture properties have the potential for strongly affecting the extension of small surface flaws during overcooling transients.

Results from the HSSI studies will be integrated to aid in resolving major regulatory issues facing the NRC. Those issues involve RPV irradiation embrittlement such as pressurized-thermal shock, operating pressure-temperature limits, low-temperature overpressurization, and the specialized problems associated with low upper-shelf (LUS) welds. Together, the results of these studies also provide guidance and bases for evaluating the overall aging behavior of LWR pressure vessels.

The program is coordinated with those of other government agencies and the manufacturing and utility sectors of the nuclear power industry in the United States and abroad. The overall objective is the quantification of irradiation effects for safety assessments of regulatory agencies, professional code-writing bodies, and the nuclear power industry.

The program is broken down into 1 task responsible for overall program management and 13 technical tasks: (1) program management, (2) fracture toughness (K_{Ic}) curve shift in high-copper welds, (3) crack-arrest toughness (K_{Ia}) curve shift in high-copper welds, (4) irradiation effects on cladding, (5) K_{Ic} and K_{Ia} curve shifts in LUS welds, (6) annealing effects in LUS welds, (7) irradiation effects in a commercial LUS weld, (8) microstructural analysis of irradiation effects, (9) in-service aged material evaluations, (10) correlation monitor materials, (11) special technical assistance, (12) Japan Power Demonstration Reactor (JPDR) steel

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examination, (13) technical assistance for Joint Coordinating Committee on Civilian Nuclear Reactor Safety (JCCCRS) Working Groups 3 and 12, and (14) additional requirements for materials. Accordingly, the chapters of this progress report correspond to these 14 tasks.

During this period, seven program briefings, reviews, or presentations were made by the HSSI staff during program reviews and visits with NRC staff or others. Six technical papers¹⁻⁶ were published, and three foreign trip reports⁷⁻⁹ issued. In addition, 18 technical presentations were made.⁹⁻²⁶

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2. K_{Ic} Curve Shift in High-Copper Welds

R. K. Nanstad

The objectives of the Fifth Irradiation Series are to determine the K_{Ic} curve shifts and shapes for two irradiated high-copper, 0.23 and 0.31 wt %, submerged-arc welds (SAWs [72W and 73W, respectively]. All planned unirradiated and irradiated testing for Phase I of the Fifth Irradiation Series has been completed. The results from statistical analyses and curve fitting of the fracture toughness results, including specimen size effects and effects of precleavage stable ductile tearing, have been reported in *Irradiation Effects on Fracture Toughness of Two High-Copper Submerged-Arc Welds, HSSI Series 5*, NUREG/CR-5913 (ORNL/TM-12156/V1), published in October 1992.

The objective of Phase II of this series is to obtain postirradiation fracture toughness data to a neutron fluence of 5×10^{19} neutrons/cm² (>1 MeV). Archive material is available and preparations are under way for machining of test specimens for welds 72W and 73W. Fracture toughness, 1TC(T), CVN, and tensile specimens will be irradiated at 288°C (550°F) to 5×10^{19} neutrons/cm² (>1 MeV) to evaluate the K_{Ic} curve shift at high fluence. The detailed specimen matrix will be dependent on final design of the irradiation facility and capsules currently under way.

3. K_{Ia} Curve Shift in High-Copper Welds

S. K. Iskander, R. K. Nanstad, and E. T. Manneschmidt

The objective of the HSSI Sixth Irradiation Series (for brevity, the K_{Ia} Program) is to determine the effect of irradiation on the shift and shape of the K_{Ia} vs $(T - RT_{NDT})$ curve, where K_{Ia} is the value of the crack-arrest fracture toughness for a crack that arrests under conditions of crack front plane-strain, T is the test temperature, and RT_{NDT} is the reference nil-ductility transition temperature, determined in accordance with Subarticle NB-2330 of *ASME Boiler and Pressure Vessel Code, Sect. III*.

3.1 Results of Testing the Sixth Irradiation Series CVN Specimens

Correlating the transition temperature shift of irradiated crack-arrest specimens to that of CVN specimens is one of the important objectives of the Sixth Irradiation Series. The determination of RT_{NDT} , the transition temperature at the 41-J impact energy level (TT_{41-J}), and its shift is based on a large number of DWT and CVN specimens that were tested as part of the Fifth Irradiation Series. In order to make some judgement about the possible differences in shift between the Fifth and Sixth Series, 22 CVN specimens were included in each of the two Sixth Series capsules. These 44 CVN specimens were recently tested. The exposures of CVN specimens were such that they were tested in two groups from each of the 72W and 73W welds. A "high fluence" group consisted of seven specimens with an average fluence of approximately 1.8 to 1.9×10^{19} neutrons/cm 2 (>1 MeV), which is an exposure level comparable to that of the crack-arrest specimens.* These seven CVN specimens, from each of the two welds, were tested to provide a better estimate of the adjusted reference temperature of the irradiated crack-arrest specimens. A "low fluence" group consisted of 15 specimens with an average fluence of approximately 1.2 to 1.3×10^{19} neutrons/cm 2 (>1 MeV) and were tested to provide data at a lower fluence level.

The results of CVN impact energy testing of the Sixth Series specimens, together with those of the Fifth Series CVN, have been plotted in Figures 3.1(a) and (b) for welds 72W and 73W, respectively. A comparison of the TT_{41-J} at the three fluence levels shows that the TT_{41-J} from the Sixth Series capsules is higher than the TT_{41-J} from the Fifth Series capsules. For the higher fluence specimens, this was to be expected but not for the lower fluence ones. To determine whether scatter or the smaller number of specimens in the Sixth Series could account for this discrepancy (56 specimens were tested in the Fifth Series), a statistical analysis has been performed and the mean TT_{41-J} and temperature span of the 95% confidence intervals on the mean for irradiated 72W and 73W welds are shown in Figure 3.2, which indicates that there is some inconsistency in the results of testing the Sixth Series CVN specimens. This matter will be investigated further and, if resolved, the reasons for this anomaly given in the final report on the Sixth Irradiation Series on crack-arrest specimens.

3.2 Results of Testing the Sixth Series Irradiated Duplex Crack-Arrest Specimens, Phase II

There were 36 weld-embrittled and 24 duplex-type specimens irradiated for the K_{Ia} Program. The 36 weld-embrittled specimens have been tested in Phase I of the K_{Ia} Program, and a detailed report has been published.¹ A summary of the objectives of the program, the materials, the specimens used, and the results have also been reported.^{2,3} In Phase II of the K_{Ia} Program, 24 duplex-type crack-arrest specimens were tested and a detailed NUREG report has been prepared.⁴

*The irradiation exposure of the Fifth Series CVN specimens is 1.5×10^{19} neutrons/cm 2 (>1 MeV).

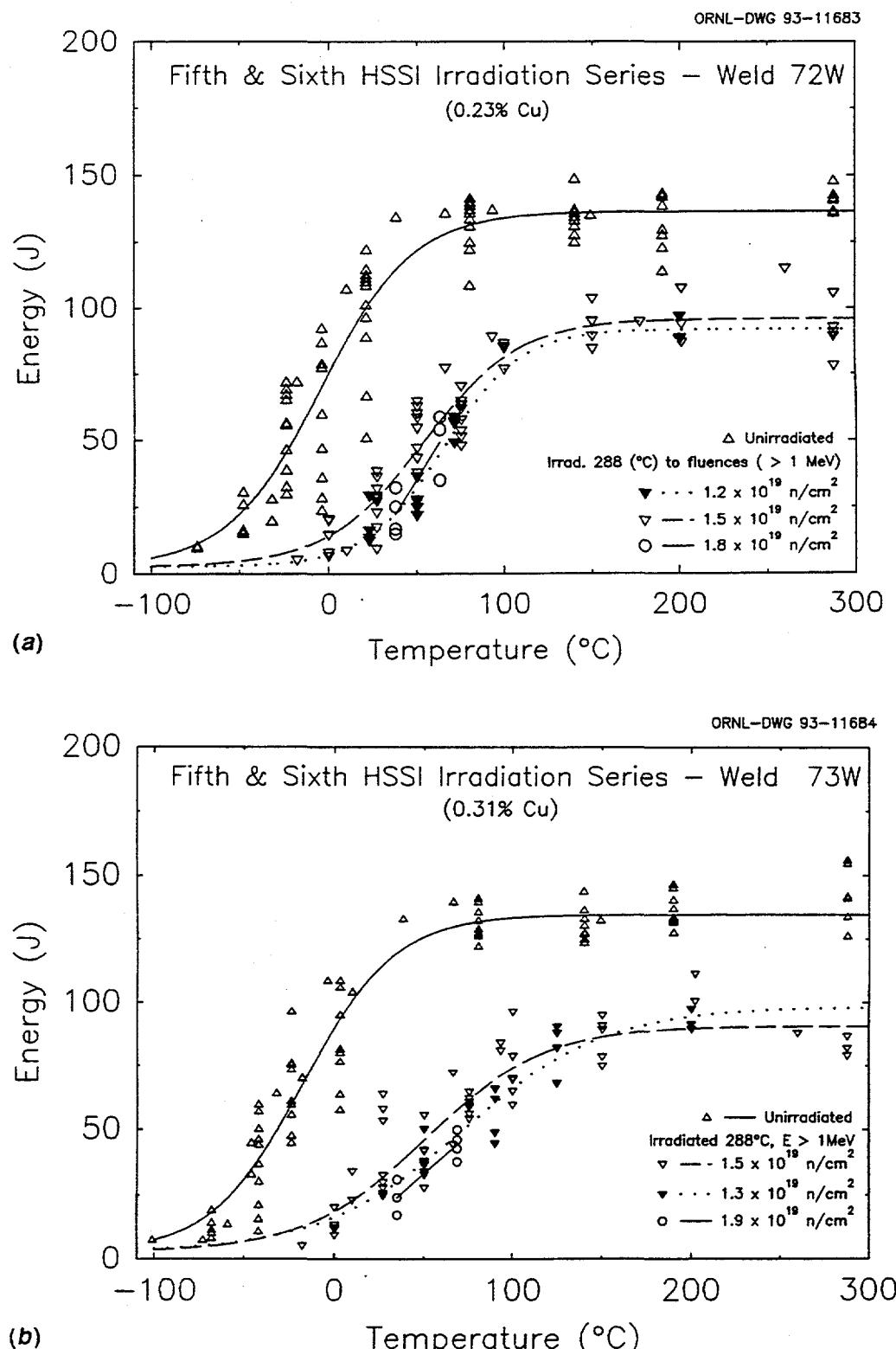


Figure 3.1. Charpy V-notch impact energy of irradiated specimens from the Fifth and Sixth Irradiation Series for (a) weld 72W and (b) weld 73W.

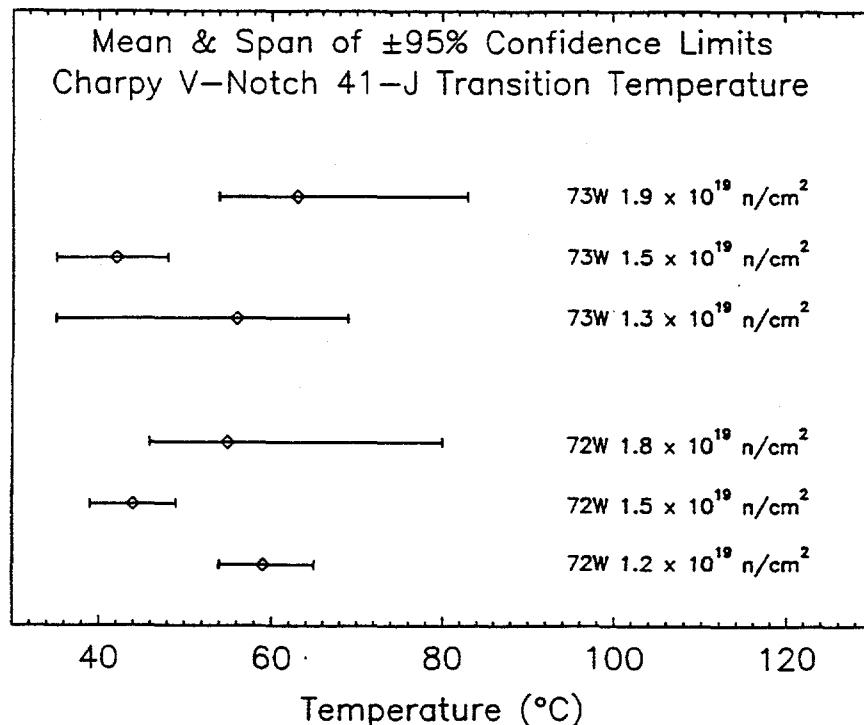


Figure 3.2. The mean TT_{41-J} and temperature span of the 95% confidence intervals on the mean for irradiated 72W and 73W welds.

The crack-arrest toughness values (K_a) for the duplex crack-arrest specimens, together with those of the weld-embrittled specimens previously obtained,¹ have been plotted against the test temperature in Figure 3.3. It may be seen that the K_a values of the duplex crack-arrest specimens all fall near the upper end of the scatter band of the K_a values of the previously tested 18 weld-embrittled crack-arrest specimens. It should be noted, however, that the average fluence of these four duplex crack-arrest specimens, 1.56×10^{19} neutrons/cm² (>1 MeV), is somewhat lower than that of the weld-embrittled crack-arrest specimens, 1.88×10^{19} neutrons/cm² (>1 MeV). Thus, the toughness values obtained from these specimens being somewhat higher than those of the weld-embrittled specimens seems reasonable.

The experimentally obtained K_a values were fitted with an equation of the form:

$$K_a = 29.4 + 1.344 \exp [0.0261 (T - T_0 + 89)] , \quad (3.1)$$

where K_a is the crack-arrest toughness in MPa \sqrt{m} ; T is the test temperature in °C; and T_0 is an unknown parameter, in °C. This equation is of the same form as the American Society of Mechanical Engineers (ASME) K_{Ia} equation.* The process was performed once with the 18 weld-embrittled crack-arrest toughness values

*In the 1992 addenda (issued Dec. 31, 1992) of the ASME Boiler Pressure and Vessel Code, the following equation (converted to SI units) for K_{Ia} is given in Article A-4000 of Sect. XI:

$$K_{Ia} = 29.4 + 13.675 \exp [0.0261 (T - RT_{NDT})] .$$

The equation appears to be the simplification of the one given in WRC Bulletin 175 (August 1972) and not that of the K_{IR} equation given in Article G-2000 of Sect. III.

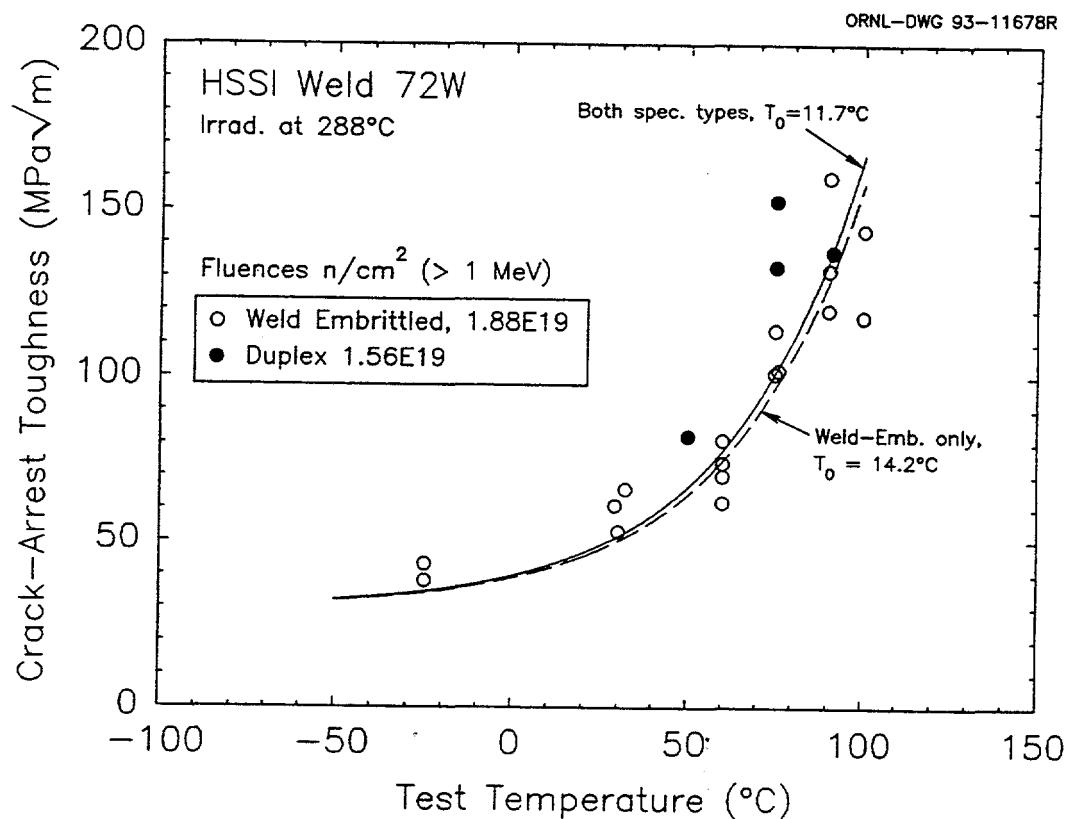


Figure 3.3. Crack-arrest toughness, K_a , for irradiated HSSI weld 72W showing the results of both weld-embrittled and duplex-type specimens.

obtained previously,³ then a second time with the results of both the 18 weld-embrittled and 4 duplex crack-arrest specimens. The T_0 s were 14 and 12°C for the weld-embrittled and both specimen types, respectively. The smaller T_0 value reflects the influence of the higher K_a values of the duplex crack-arrest specimens compared to those of the weld-embrittled specimens. It may be seen that the results from the duplex crack-arrest testing have not made a significant difference to the results of testing the weld-embrittled specimens.

The experimentally obtained crack-arrest toughness values for both unirradiated and irradiated 72W weld metal and for weld-embrittled and duplex-type specimens are plotted in Figure 3.4. Also shown on the same figure are two curves with a T_0 determined by fitting Equation (3.1) to the K_a data for the 72W weld. The shift between the two curves is 84°C, within 1° of that for the CVN specimens irradiated to 1.8×10^{19} neutrons/cm² (> 1 MeV). It should be recalled that the four duplex crack-arrest specimens were only irradiated to 1.5×10^{19} neutrons/cm² (> 1 MeV) compared to 1.9×10^{19} neutrons/cm² (> 1 MeV). No adjustment was made when they were considered as one set with the weld-embrittled specimens. Such adjustments may be made in the final report on the Sixth Series.

3.3 Preparations for Testing Irradiated Crack-Arrest Specimens Supplied by ENEA

A new remote crack-arrest fixture for testing the large irradiated specimens from the Italian Committee for Research and Development of Nuclear Energy and Alternative Energies (ENEA) has been built and used to test crack-arrest specimens from the Midland weld (see Task 7). Preliminary indications are that the temperature distribution in the new fixture is not as uniform as in the previous fixture, probably because of the

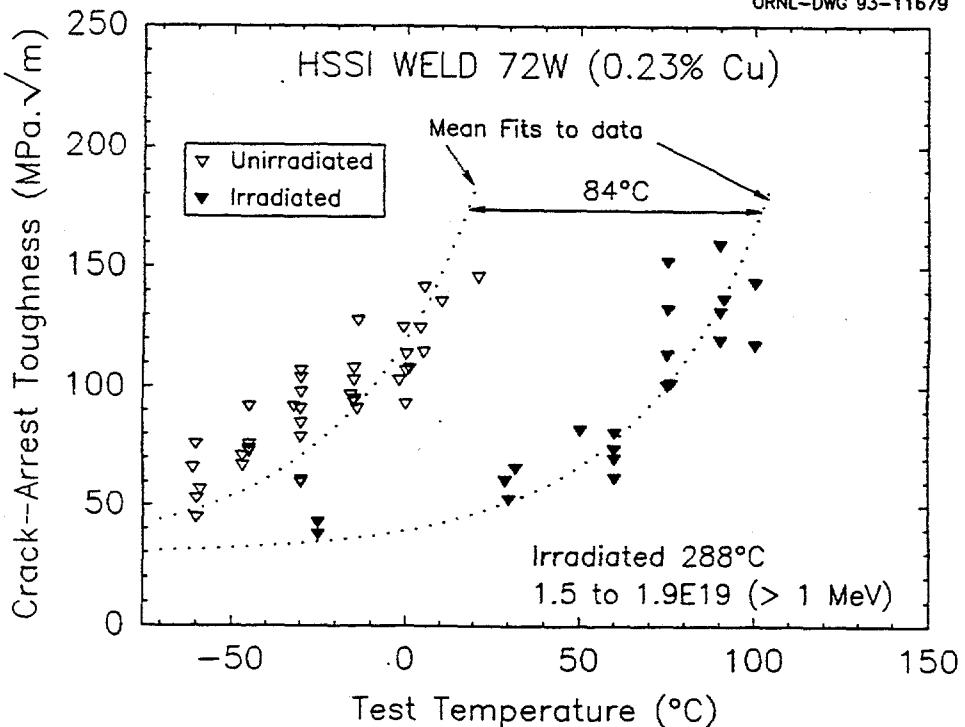


Figure 3.4. Crack-arrest toughness values for both unirradiated and irradiated 72W weld metal and for weld-embrittled and duplex-type specimens.

larger size. The impetus for building the new fixture is that the NRC has agreed to test the irradiated crack-arrest specimens for ENEA at ORNL. The results will have usefulness and applicability to the safety assessment of U.S. RPVs. The temperature distribution of the various sized specimens in both normal and "inverted" positions in the new fixture still needs to be determined, and a method to measure the crack-mouth opening of the ENEA crack-arrest specimens must be devised. Some background information on the ENEA program has been given in the previous semiannual report.

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* Available for purchase from National Technical Information Service, Springfield, VA 22161.

† Available in public technical libraries.

4. Irradiation Effects in Cladding

F. M. Haggag

The objective of this series is to obtain toughness properties of stainless steel cladding in the unirradiated and irradiated conditions. The properties obtained include tensile, CVN impact, and J-integral toughness. The goal is to evaluate the fracture resistance of irradiated weld-metal cladding representative of that used in early pressurized-water reactors. The fracture properties are needed for detailed integrity analyses of vessels during overcooling situations. There was no significant activity within this task during this reporting period.

5. K_{Ic} and K_{Ia} Curve Shifts in LUS Welds

R. K. Nanstad, D. E. McCabe, and S. K. Iskander

The objectives of the HSSI Eighth Irradiation Series are to evaluate the irradiation-induced temperature shifts and shape changes of the K_{Ic} and K_{Ia} curves for high-copper, low CVN upper-shelf welds. These objectives are similar to those of the Fifth and Sixth Irradiation Series,^{1,2} which were conducted with high-copper SAWs with relatively high CVN upper-shelf energies (USEs). The welds of the Fifth Series, designated 72W and 73W, contained copper contents of 0.23 and 0.31 wt %, respectively, and gave CVN USEs of about 135 J (100 ft-lb) in the unirradiated condition. A number of SAWs fabricated by Babcock and Wilcox with Linde 80 welding flux and copper-coated welding wire exhibit CVN USEs of about 100 J (75 ft-lb) and less in the unirradiated condition. The low USEs of these welds derive from the very high number of small, nonmetallic inclusions contained in the welds. Furthermore, because of the relatively high copper contents, many of those welds exhibit relatively high irradiation-induced CVN transition temperature shifts and USE decreases.

The previous semiannual report presented discussion regarding use of the weld wire used to fabricate HSSI weld 73W (0.31% Cu). Combined with Linde 80 welding flux, a weld with low Charpy USE and a copper content of about 0.31% should result. Sufficient weld wire likely exists for conduct of this program (the test matrix has not been finalized), but the archival supply would probably be depleted, and other options are under discussion.

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* Available for purchase from National Technical Information Service, Springfield, VA 22161.

6. Annealing Effects in LUS Welds

**S. K. Iskander, M. A. Sokolov, R. K. Nanstad,
R. E. Stoller, and E. T. Manneschmidt**

The purpose of the Ninth Series is to evaluate the correlation between fracture toughness and CVN impact energy during irradiation, annealing, and reirradiation (IAR).

Eighteen so-called "undersize" irradiated CVN specimens of HSSI 73W weld metal were annealed and tested. The 41-J energy level transition temperature, TT_{41-J}, of the irradiated/annealed (I/A) undersize CVN specimens has recovered approximately 90%, and the USE has recovered 125%. Background information and a summary of results were presented in the previous semiannual. The USE of I/A CVN specimens is approximately 25% greater than the unirradiated USE. To determine if this increase in USE is due to thermal aging, unirradiated specimens were aged in air at 460 and 490°C (860 and 915°F) for 168 h. The results of testing these aged specimens are given below.

The USE of the specimens aged at 460 and 490°C for 168 h are given in Table 6.1 and compared to the USE of unirradiated and I/A specimens. It appears that aging does account for most of the increase exhibited by the irradiated and aged specimens. The USE of the I/A specimens is still about 7% higher than that of the aged specimens, but considering the scatter on the upper shelf, the difference may not be significant.

Unirradiated CVN impact specimens from the Russian VVER-440 weld metal have been aged at 460 and 490°C for 168 h. These tests showed that aging of unirradiated RPV welds can increase the value of absorbed energy in the upper-transition region as well as the USE (see Figure 6.1). This weld contains about 0.030% P, which may represent high-phosphorus welds of the oldest VVER-440 reactor vessels.

The increase in the USE of I/A welds above the unirradiated level is not surprising since it has been observed by others.¹ However, the USE increase in the unirradiated material following annealing for 168 h at 460 and 490°C was not expected. The HSSI 73W weld had received a postweld heat treatment (PWHT) of 607°C (1125°F) for at least 40 h, and the VVER-440 weld had also received a PWHT, but at 670°C (1240°F) for 34 h. Thus, it seems unlikely that relatively short-term aging at a lower temperature would significantly alter the microstructure. It is instructive to use a time-temperature relationship² to estimate the annealing response in

**Table 6.1. Effects of irradiation, annealing, and aging
on Charpy upper-shelf energy of undersize
HSSI weld 73W specimens**

	Upper-shelf impact energy (J)	Change ^a (%)
Unirradiated	117 ^b	0
Irradiated and annealed ^c at 454°C	147 ^b	26
Aged ^c at 460°C	138 ^d	18
Aged ^c at 490°C	138 ^d	18

^aBased on upper-shelf energy of unirradiated specimens.

^bObtained from a hyperbolic tangent fit and/or the average of specimens tested at 150°C.

^cAged for 168 h.

^dAverage of four specimens tested at 150°C.

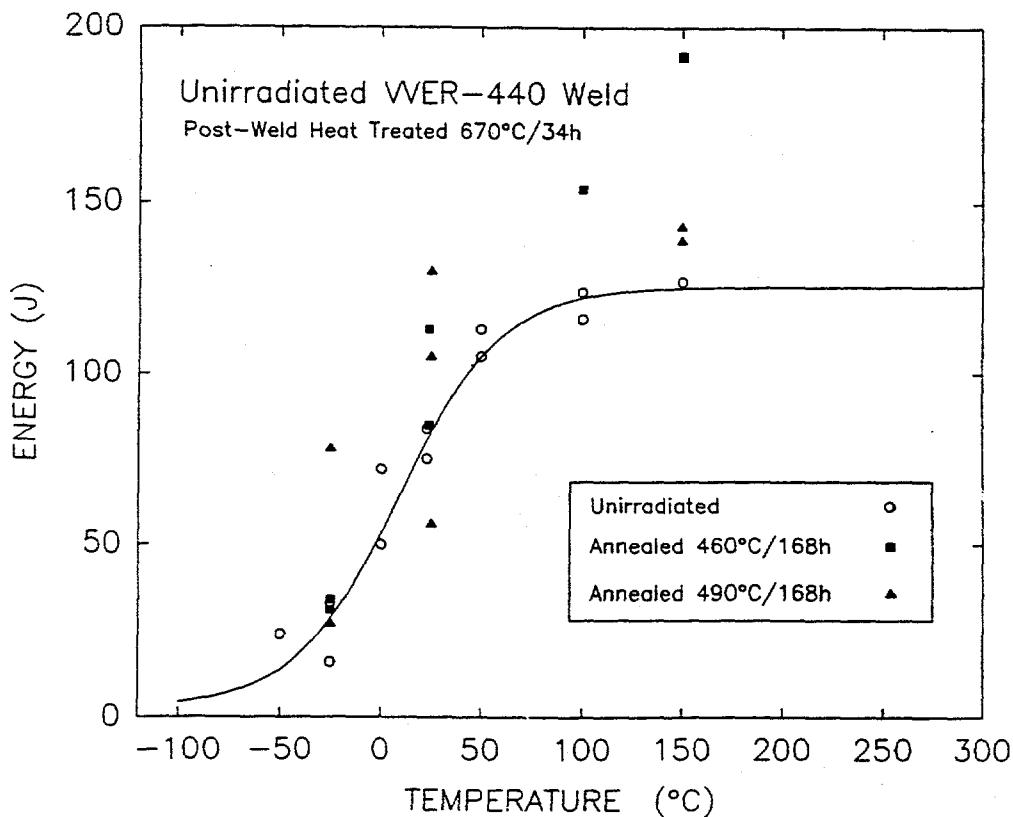


Figure 6.1. Charpy V-notch impact energy of unirradiated VVER-440 weld metal, containing 0.030% P, in the unirradiated and unirradiated/aged conditions.

terms of the increment in time at 607°C that would correspond to the annealing at 460°C for 168 h. One of the time-temperature relationships, the Hollomon-Jaffee, giving the so-called annealing parameter, TP , can be rewritten in the following form:

$$TP = T \log (10^c \cdot t) \times 10^{-3} , \quad (6.1)$$

where T is temperature in kelvin, t is the time in hours, and c is an experimentally determined constant for carbon steel welds that has been observed to vary between 10 and 20 (ref. 2). The time at 607°C that would give an annealing "equivalent" in terms of the effect on the impact properties to an annealing given at 460°C for 168 h is about 1 h for $c = 10$ and about 1 min for $c = 20$. This would not make much difference to material that was annealed for 40 h. Hence, the time-temperature concept does not help in explaining the observed effects of annealing the unirradiated material. In the case of the VVER-440 weld, because of the higher PWHT temperature, the lower temperature anneal would give an even less time contribution.

Additionally, the response of the unirradiated material to the 168-h anneal would be expected to be different from the irradiated weld since the starting microstructures would be quite different. The unirradiated material would not contain the fine dispersion of radiation-induced precipitates and point defect clusters that are responsible for the initial ductile-to-brittle temperature transition (DBTT) shift. Plans are to examine the unirradiated material in both the as-fabricated and annealed conditions to determine whether a microstructural basis for the increase in the USE exists.

The CVN impact tests were performed at a temperature of 150°C to determine the effect of annealing in air at 460 and 490°C for 168 h. There were also four specimens tested at -44 and 36°C to determine the effect of annealing in air on the impact energy in the lower- and upper-transition regions, where the surface condition of the notch is expected to affect crack-initiation toughness and, hence, the impact energy. Figure 6.2 gives the impact energy values obtained from testing undersized CVN specimens from 73W weld metal in the following conditions:^{*} I/A, unirradiated, and unirradiated and annealed at two temperatures. It appears that the annealing at the temperatures and times mentioned above did not have a significant effect on the impact energy on either of the four unirradiated specimens tested in the lower or upper transition regions.

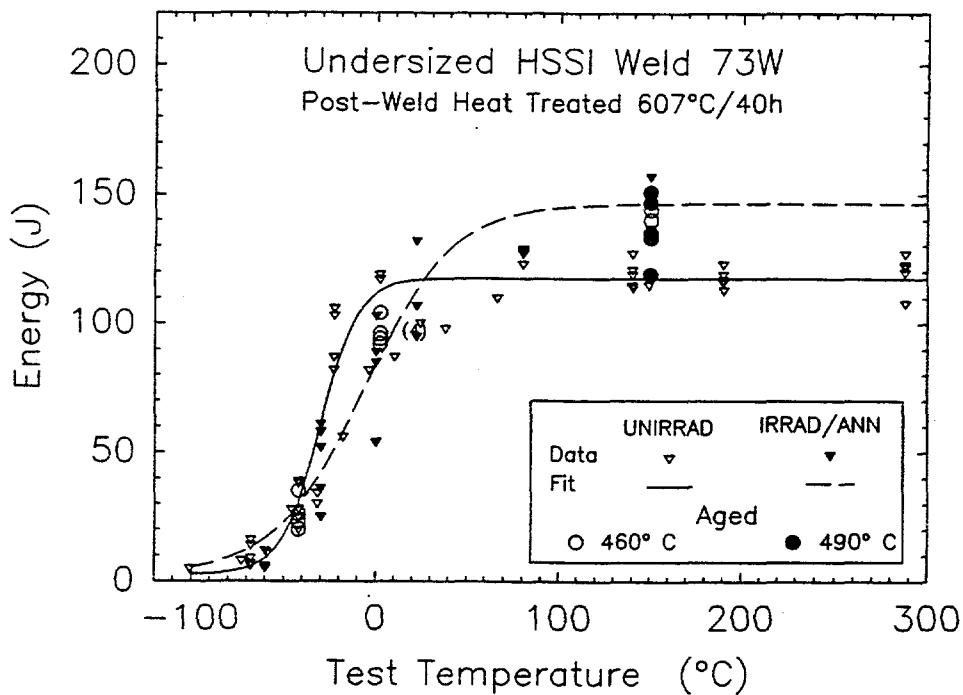


Figure 6.2. Charpy V-notch impact energy of undersize specimens of HSSI weld 73W weld metal in the unirradiated, unirradiated/aged, and irradiated/annealed conditions.

References

1. J. R. Hawthorne, Materials Engineering Associates, Inc., *Irradiation-Anneal-Reirradiation (IAR) Studies of Prototypical Reactor Pressure Vessel Weldments*, USNRC Report NUREG/CR-5469 (MEA-2364), November 1989.[†]
2. R. K. Nanstad, G. M. Goodwin, and M. J. Swindeman, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., *Effects of Nonstandard Heat Treatment Temperatures on Tensile and Charpy Impact Properties of Carbon-Steel Casting Repair Welds*, USNRC Report NUREG/CR-5972 (ORNL/TM-12280), April 1993.[†]

^{*}The comparison of the impact energy of full to undersized and unirradiated to I/A CVN specimens was presented in the previous semiannual report.

[†]Available for purchase from National Technical Information Service, Springfield, VA 22161.

7. Irradiation Effects in a Commercial LUS Weld

D. E. McCabe and S. K. Iskander

7.1. Results of Testing Crack-Arrest Specimens of Midland Weld Material

Thirty weld-embrittled crack-arrest specimens were prepared from the Midland weld, half of which are being irradiated. This section presents the results of tests on the unirradiated specimens.

The results of crack-arrest tests on the Midland weld material obtained are given in Table 7.1 and Figure 7.1. The weld electrode used to prepare the brittle crack-starter bead, McKay DWT, was the same as that used to prepare the brittle crack-starter weld bead for DWT specimens. The experience with these weld electrodes was not satisfactory, as it is believed that the heat-affected zone created by these electrodes may be tougher than necessary. As a result, considerable tearing through that zone has to occur before a fast-running crack initiates. Moreover, it appears that the size of the specimen was somewhat too small as may be seen from Table 7.1. Thus, the remaining ligament in most of the tests was smaller than that recommended by American Society for Testing and Materials (ASTM) E 1221-88. Nevertheless, it appears that the results are reasonable estimates of valid K_a .

Table 7.1. Crack-arrest toughness values, K_a , of submerged-arc weld from the beltline region of the Midland reactor pressure vessel measured using specimens with a nominal width of 104 mm, except for specimen MW15JC, with a nominal width of 127 mm

Specimen	Test temperature (°C)	Crack-arrest toughness, K_a (MPa \sqrt{m})	Validity* and comments
MW12A1B	-40	58.5	a,b
MW12EBB	-40	75.3	a,b,e
MW12A1	-30	76.3	a,b,e
MW12D1A	-30	78.7	a,b,e
MW12HBB	-30	91.8	a,b,e
MW12EAB	-30	93	a,b
MW12GAB	-25	92.9	a,b
MW15JC	-20	65.3	Valid, 150-mm spec
MW15HAA	-20	101.1	a,b
MW12FBB	-20	148.4	a,b,c,e
14DRW34	-10	107.5	a,b,e
MW12HBA	0	90	a,b
MW12HAA	10	95.4	a,b,e

One or more letters for a specimen indicate that the test results did not meet one of the minimum lengths of the ASTM E 1221-88 validity criteria. The letters correspond to those in Table 2 of ASTM E 1221-88, as follows:

a,b = remaining ligament too small.
c = specimen too thin.
d,e = insufficient crack-jump length.

Note: K_a = value of stress intensity shortly after arrest.

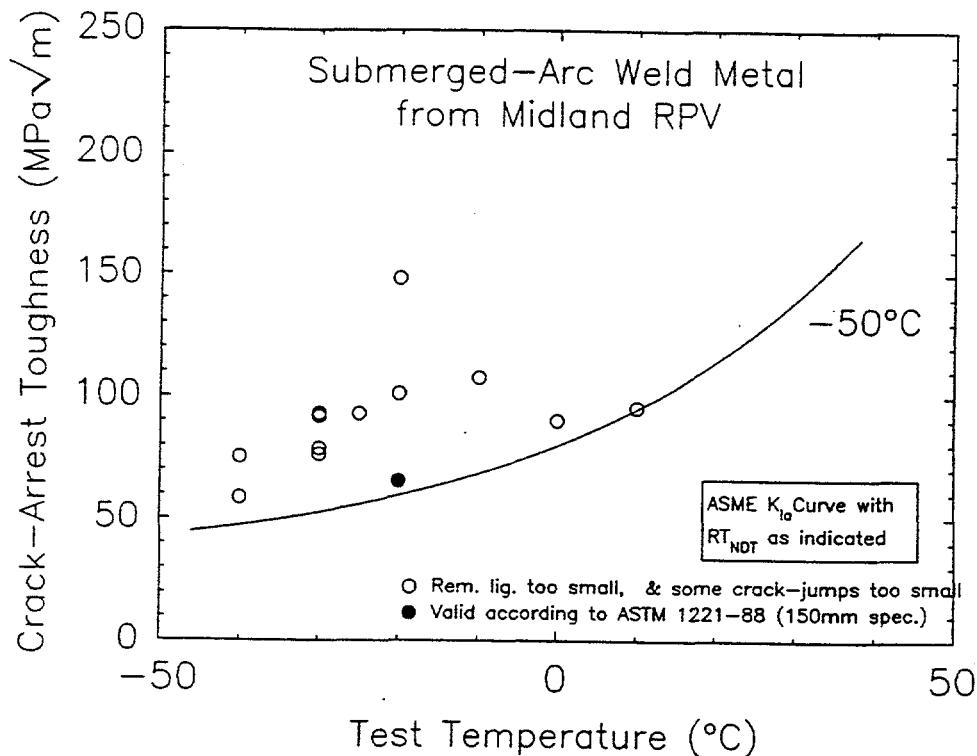


Figure 7.1. Crack-arrest toughness values, K_a , of specimens machined from submerged-arc weld of Midland reactor pressure vessel. Specimens are oriented so that the crack propagation is in the welding direction.

The preparation of a few duplex-type crack-arrest specimens was delayed because of problems with the electron-beam welder but is now progressing. The duplex-type crack-arrest specimens will be used to obtain crack-arrest toughness, K_a , at a higher temperature than can be obtained using weld-embrittled specimens. The new, remote crack-arrest fixture, destined to be installed in the hot cells for use with irradiated specimens, was used to test these unirradiated specimens as well as to test the fixture itself.

7.2. Unirradiated Testing and Evaluation

A draft NUREG report, *Unirradiated Material Properties of Midland Weld WF-70*, has been prepared. It describes the sampling plan for specimens taken from the nozzle course and beltline welds. The baseline material characterization work has been previously reported.¹ The basis for classifying nozzle course and beltline WF-70 weld metals is presented. Although the standard mechanical property evaluations of Charpy V and DWT NDT tests indicated no difference in fracture toughness property; the fracture mechanics tests clearly indicated that the nozzle course weld metal had a 27°C (49°F) higher transition temperature. Tensile tests also indicated that the nozzle weld metal had higher strength than the beltline weld metal.

J-R curves were developed at test temperatures of 21, 150, and 288°C (70, 302, and 550°F). The Charpy V/J-R curve correlation developed by Eason et al.² using a multivariable method was tested against these experimental J-R curves. The comparison was good at all test temperatures except room temperature. A tacit assumption made in the Eason model is that all specimens are side grooved. Side grooving is not a regulated variable in the ASTM J-R curve test standard, and this is potentially a way to manipulate J-R curve data to satisfy fracture toughness requirement needs. Another subject addressed is the extreme variability in RT_{NDT} associated with the determination of RT_{NDT} from Charpy transition curves. The scheme outlined in Sect. III of

the ASME Code³ was used on 19 Charpy transition curves taken from various locations along the beltline weld. The RT_{NDT} varied from +37 to -20°C (+9 to -4°F).

7.3 Material Irradiations

Scoping capsules 10.01 (beltline WF-70) and 10.02 (nozzle WF-70) were fabricated at Materials Engineering Associates and exposed to 0.5×10^{19} neutrons/cm² in the University of Buffalo Reactor, Buffalo, New York. Large capsule 10.05 (1T size) completed irradiation in July 1993 and is currently being held in the reactor pool of the Ford Nuclear Reactor at Ann Arbor, Michigan. The disassembly plans require further study. The three options are (1) disassemble at the Phoenix Laboratory, University of Michigan; (2) seek an independent hot-cell facility; or (3) transport to ORNL for disassembly. The evaluation work is in progress.

Large capsule 10.06 (2T size) has been fabricated, and the irradiation has been started. The control instrumentation equipment has been checked, calibrated, and is working satisfactorily. This capsule contains compact specimens of nozzle and beltline welds ranging in size from 1/2T to 2T, in addition to crack-arrest, tensile, and Charpy specimens. Although the major part of the capsule space contains Midland WF-70 weld materials, Russian pressure-vessel steel and weld metal from the HSSI Fifth Irradiation Series were included. This exposure will require the better part of a year to be completed at 1×10^{19} neutrons/cm².

References

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3. *ASME Boiler and Pressure Vessel Code. An American National Standard*, Sect. III, Article NB-2330, "Test Requirements and Acceptance Standards," American Society of Mechanical Engineers, New York, 1992.†

*Available for purchase from National Technical Information Service, Springfield, VA 22161.

†Available in public technical libraries.

8. Microstructural Analysis of Radiation Effects

R. E. Stoller, K. Farrell, S. K. Iskander,
S. T. Mahmood, and P. M. Rice

8.1 Trojan Cavity Irradiations

Concern has been raised by the higher-than-expected increase in the transition temperature shift of RPV steels subjected to irradiations at relatively low temperature ($\sim 50^{\circ}\text{C}$) and low flux ($\sim 10^9 \text{ n}\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$) [ref. 1]. A study into the effect of exposure to a relatively low flux on support structures (such as found in prototypical power reactor plant cavities)² has already been completed. As part of an ongoing investigation into the causes of this low-temperature embrittlement, specimens of structural steel were placed in a capsule in the middle of 1990 to be irradiated in the cavity of the Trojan nuclear reactor power plant. The specimen complement is shown in Table 8.1. The 11 CVN impact specimens were full-sized ($10 \times 10 \times 55$). The 44 mini-tensile specimens were packaged into a special holder whose external dimensions were approximately those of a single, full-sized CVN specimen. The holder is held together by tack-welded stainless steel straps; see Figure 8.1. It is designed to be easily disassembled by cutting the straps with a knife. The dimensions of the mini-tensile specimens are approximately $0.76 \times 5 \times 25 \text{ mm}$ ($t \times w \times l$) and are of a flat shape geometrically similar to that used to test sheet materials.

Table 8.1. Specimen complement enclosed in a capsule for placement in the Trojan Reactor cavity

Full-size Charpy V-notch specimens		Mini-tensile specimens ^a		
A 36	A 212 B	A 36	A 350-3	A 212 B
A364	2CO3	601	1	201
A365	2CO4	602	2	202
A366	2CO5	606	3	203
A367	2CO6	607	4	204
A368	2CO7	608	5	205
	2CO8	609	6	206
		610	8	207
		611	9	208
		612	10	209
		615	11	210
		617	12	211
		618	13	212
		619	15	213
		620	17	214
			18	215

^aAll 44 mini-tensile specimens were placed in a single container whose size is that of a full-size specimen.

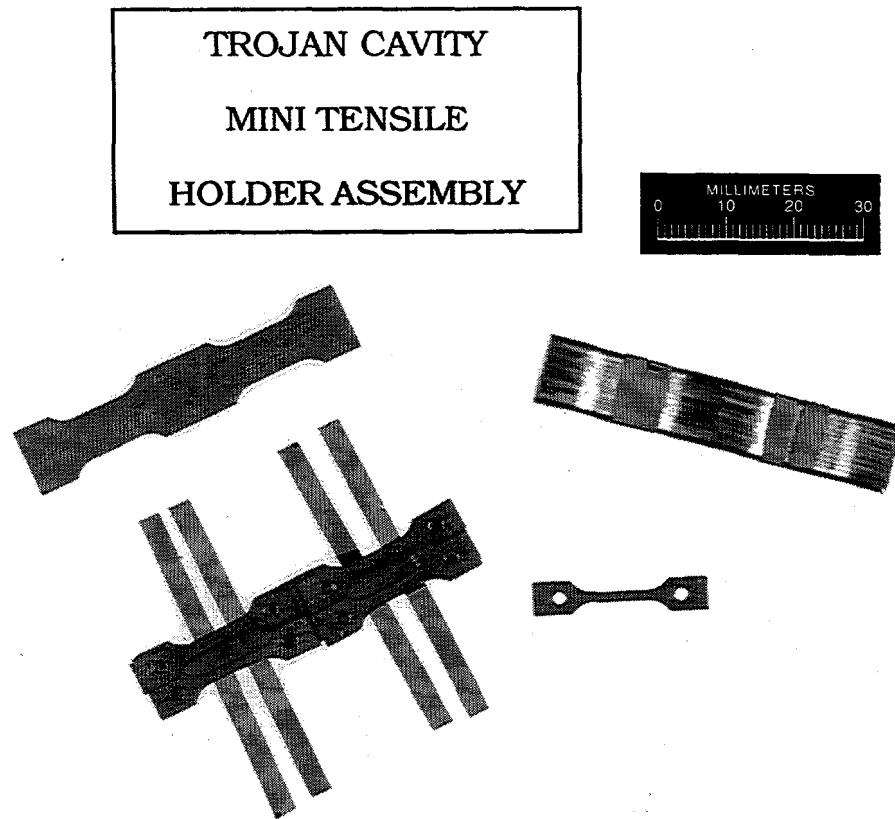


Figure 8.1. Holder for 44 mini-tensile specimens with external dimensions approximately that of a full-sized Charpy V-notch specimen.

Specimens were machined from three materials; their chemical compositions are given in Table 8.2. The A 212 B specimens were machined from a carbon steel plate conforming to ASTM A 212, *Specification for High-Tensile-Strength Carbon-Silicon Steel for Boilers and other Pressure Vessels*, Grade B (A 212 B). The other two materials were 3.5 wt % Ni forging steel conforming to ASTM A 350 LF3 and a structural steel conforming to ASTM A 36. The A 212 B and the A 350 LF3 steels were from archival material used in connection with the High Flux Isotope Reactor safety assessment study.¹

The A 212 B CVN impact specimens were tested, and the results are shown in Table 8.3. The irradiation temperature was approximately 60°C, and the estimated fluence of the specimens is approximately 1×10^{16} n/cm² (E > 1 MeV). The results were compared to unirradiated and irradiated data available in refs. 1 and 3 for A 212 for material from the same plate and are shown in Figure 8.2. The CVN impact energy values at this low fluence are indistinguishable from that of unirradiated material. This figure also illustrates the anomalously high CVN shifts observed in the HFIR surveillance program. The results of tests on the A 36 specimens are being analyzed.

8.2 Irradiation Experiment to Investigate Microstructure/Mechanical Property Correlation

Nine model alloys were acquired for use in a study of microstructure/mechanical property correlation following ion irradiation. The purpose of this experiment is to determine the dislocation barrier strength of small point defect clusters in irradiated steels. These clusters can be responsible for much of the hardening observed in

Table 8.2. Chemical composition (wt %) of ferritic materials used to machine Charpy V-notch and mini-tensile specimens

Element	A 212 B	A 350 LF3	A 36
C	0.26	0.18	0.21
Al	0.07	0.08	0.003
Co	0.015	0.03	0.007
Cr	0.075	0.09	0.04
Cu	0.15	0.11	0.05
Mn	0.85	0.55	1.1
Mo	0.02	0.03	0.03
Nb	<0.001	<0.001	?
Ni	0.09	3.3	0.07
Si	0.29	0.29	0.3
Sn	0.02	0.02	0.002
Ti	0.01	<0.001	<0.001
V	0.0005	0.001	0.001
W	<0.005	<0.005	<0.01
Zr	<0.001	<0.001	<0.001
P	0.006	0.01	0.009
S	0.04	0.02	0.026
As	0.007	0.01	0.007
B	<0.0005	<0.0005	<0.001
N	0.006	0.009	?
O	0.0024	0.0027	?

Table 8.3. Charpy V-notch impact energy of L-T orientation specimens of A 212 High Flux Isotope Reactor archival material irradiated in the Trojan cavity at 60°C to approximately 1×10^{16} n·cm $^{-2}$ (>1 MeV)

Specimen ID	Temperature		Energy		
	(°F)	(°C)	(ft-lb)	(J)	Shear (%)
2C07	20	-7	29.3	40	40
2C04	20	-7	26.5	36	25
2C08	20	-7	26.6	36	20
2C06	90	32	86.0	117	99
2C05	90	32	67.4	91	85
2C03	90	32	65.3	89	80

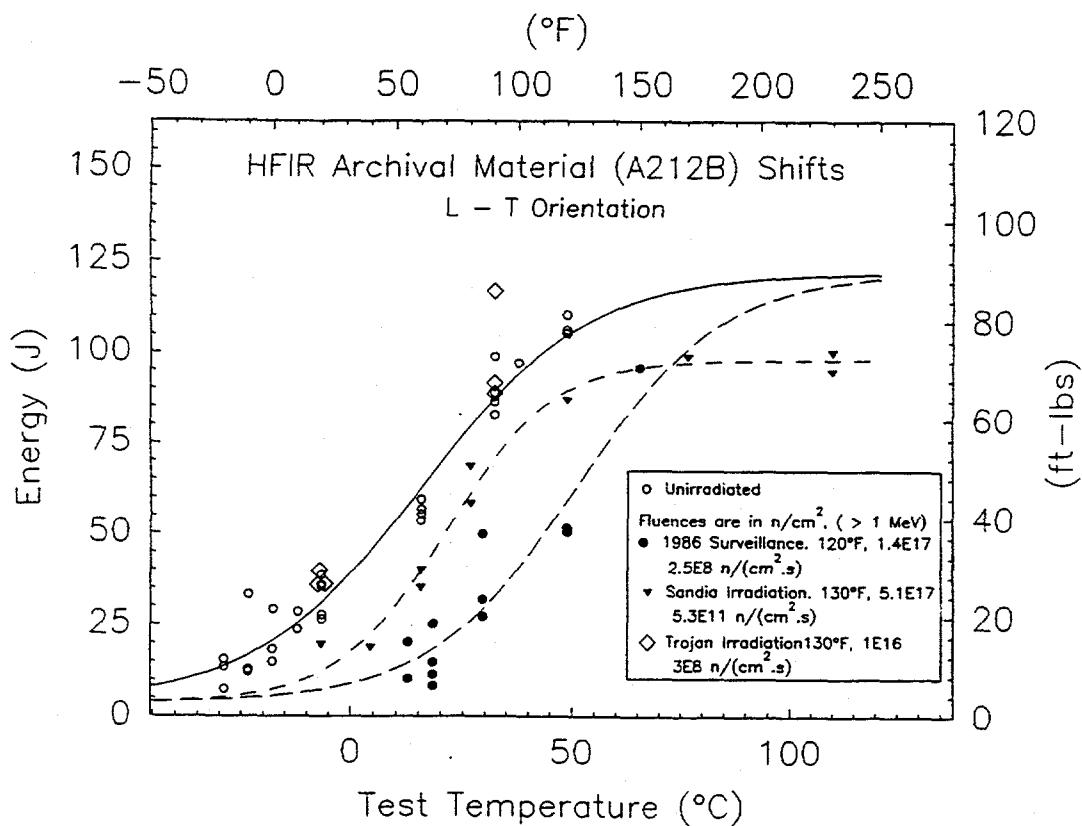


Figure 8.2. Charpy V-notch impact energy of L-T orientation specimens subjected to low-temperature irradiation at high and low flux.

these materials.⁴ The materials were obtained from the University of California, Santa Barbara, in the form of 0.020-in.-thick sheet. These alloys are a subset of those being used in a large neutron irradiation experiment to investigate the effects of chemical composition, displacement rate (neutron flux), and neutron spectrum on low-temperature embrittlement.

A number of developmental problems have been solved in order to obtain acceptable specimens for use in the TEM, including the plating of nickel on the steel specimens to limit the amount of magnetic ferritic material in the TEM when examining cross-sectioned specimens. The preliminary TEM examination of the model alloys in the as-received condition has been completed. One of the high-copper alloys has been heat-treated to artificially harden the material by the formation of copper precipitates. The base iron with low and high nitrogen contained a fairly high dislocation density and iron-nitride precipitates with sizes up to about 1 μm . The Fe-0.9 Cu exhibited a number of Cr-Mn-containing precipitates, apparently due to residual impurities. No copper precipitates were observed in the as-received condition. After annealing for 16 h at 550°C, a high density of 8- to 10- nm copper precipitates was observed by high-resolution TEM. The copper precipitates displayed the expected 9R structure.

The first ion irradiation experiments have been completed. The materials included in these experiments were the low-nitrogen pure iron and the low-nitrogen Fe-0.5Cu alloy. Data from these irradiations will provide a low- and high-copper baseline for comparison with the remaining alloys that will be irradiated later. These irradiations were conducted using 4-MeV iron ions at temperatures of 300 and 400°C to doses of 0.01, 0.1, and 1.0 displacements per atom (dpa). The displacement rate was about 7.0×10^{-5} dpa/s. This is much higher than a typical displacement rate for LWR pressure vessels, which is 10^{-11} to 10^{-10} dpa/s. The higher irradiation temperature was included in this experiment due to this accelerated displacement rate. Previous

work has demonstrated the relationship between irradiation temperature and displacement rate, with the "effective" irradiation temperature increasing as the displacement rate increases.

8.3 Microstructural Modeling and Analysis

An extensive set of molecular dynamics (MD) calculations has been carried out to investigate the evolution of displacement cascades in iron. This work is being carried out in collaboration with staff at the Harwell Laboratory (W. J. Phythian and A. J. E. Foreman) and the University of Liverpool (D. J. Bacon and A. F. Calder) in the United Kingdom. Cascades with energies between 60 eV and 10 keV have been simulated for temperatures in the range of 100 to 900 K. The MD simulation energy is essentially equivalent to the damage energy in the standard NRT displacement model.⁵ A 10 keV MD simulation energy is equivalent to a primary knock-on atom (PKA) energy of 13.7 keV in iron. The results have been compared to earlier work that examined displacement cascades in copper to determine whether there were any systematic differences between the face-centered-cubic material (copper) and the body-centered-cubic (bcc) material (iron).

A typical result of this comparison is shown in Figure 8.3 where the number of stable Frenkel pairs from the MD calculation at 100 K is normalized to the number of displacements calculated from the standard NRT model.⁵ For the conditions shown in Figure 8.1, the number of point defects has stabilized by about 10 ps. The dependence of this ratio on PKA energy is similar in iron and copper, but a greater number of point defects survive in the more open bcc structure of iron. Very little temperature dependence is observed between 100 and 900 K. The fact that the MD/NRT dpa ratio is greater than 30% for a 13.7-keV PKA is significant since some researchers have suggested that this ratio could be as low as a few percent for fast neutrons. At steady state, the vacancy concentration is directly proportional to this ratio. The use of the proposed lower values in modeling studies would lead to an underprediction of the point defect cluster population and of the radiation-enhanced diffusion coefficient that drives copper precipitation in pressure-vessel steels.

Another significant difference between the two materials is that the fraction of the surviving point defects that are contained in clusters is lower in iron than in copper. In particular, essentially no vacancy clusters are observed in iron. The fraction of clustered point defects is also an important parameter in the kinetic model that is being developed to predict embrittlement as will be discussed below. It is possible that vacancy clusters will be observed for higher PKA energies, since the present calculations do not sample the complete neutron energy spectrum. The highest PKA energy simulated is 13.7 keV, which is the average value for a 0.4-MeV neutron. However, since this is well into the fast part of the spectrum, it is likely that the importance of in-cascade vacancy cluster formation will be modest. An example of the interstitial clustering behavior is given in Table 8.4 for 10-keV cascades at 100 K. This table also illustrates the statistical variation between typical cascades.

The results of the MD simulations were used to refine cascade survival and clustering parameters in the point defect clustering model that is being developed under this task. Several other revisions were made to update the model, and a detailed series of calculations were carried out to investigate the dependence of the model's predictions on a number of material and irradiation parameters. A detailed discussion of the results obtained with the revised model was presented at the "Sixth International Symposium on Environmental Degradation of Materials in Nuclear Power Systems: Water Reactors" and will be published in the proceedings of this meeting. The most significant observation in these new calculations comes out of the MD calculations which indicated that little in-cascade vacancy clustering occurs for PKA energies up to 13.7 keV. This significantly reduces the hardening contribution of vacancy clusters relative to interstitial clusters. The calculated change in the yield strength at 288°C for a dose of 0.01 dpa is reduced by essentially an order of magnitude for RPV operating conditions and more than a factor of two for test reactor irradiation conditions. Parameter choices that influence interstitial clustering take on new importance as a result of the potentially increased significance of interstitial clusters in hardening. The binding energy of interstitials in small interstitial clusters was identified as a critical parameter in the model, and a method of investigating this parameter via additional MD work will be pursued.

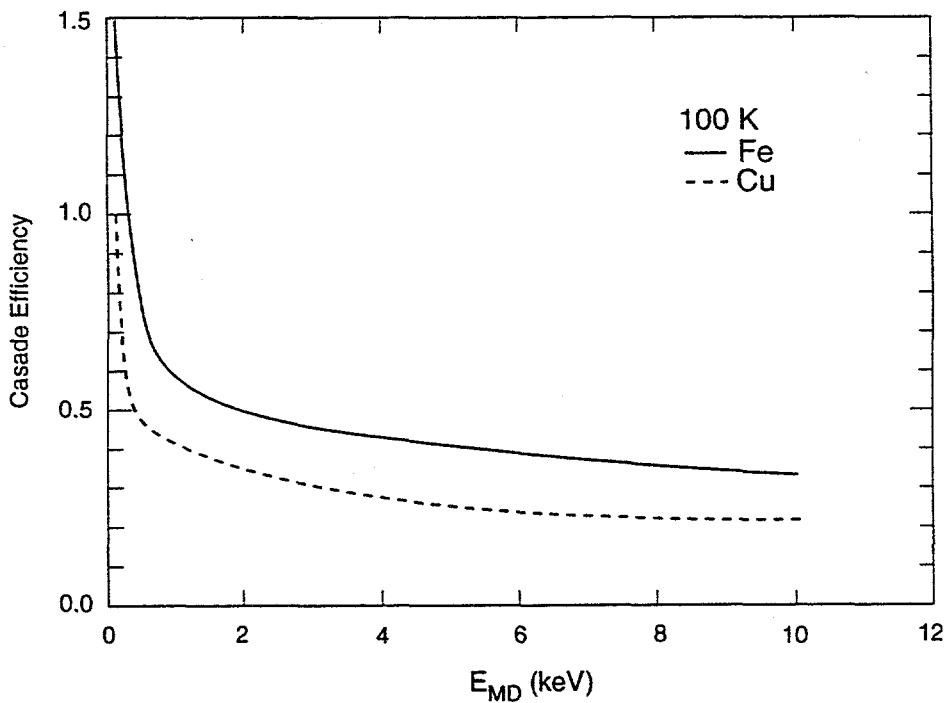


Figure 8.3. Ratio of MD stable point defects (>10 ps) to NRT dpa in iron and copper as a function of MD cascade energy.

Table 8.4. Summary of point defect and interstitial cluster production from 10-keV MD simulations (13.7 keV PKAs) at 100 K

Cascade No.	Frenkel pair at >10 ps	Cascade efficiency ^a	Number of interstitial clusters				
			2-i	3-i	4-i	5-i	8-i
1	37	0.37	5	-	1	-	-
2	28	0.28	3	1	-	-	-
3	34	0.34	2	2	-	-	-
4	34	0.34	4	1	1	-	-
5	31	0.31	-	3	-	1	-
6	37	0.37	2	1	1	-	-
7	32	0.32	2	-	-	-	1
Average	33	0.33	2.6	1.1	0.42	0.71	0.14

^aCascade efficiency is defined as the number of residual point defects divided by the calculated NRT dpa (100 for a damage energy of 10-keV with a 40-eV displacement energy).

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5. "Standard Practice for Neutron Radiation Damage Simulation by Charged Particle Irradiation," ASTM E 521-89 in *Nuclear (II), Solar, and Geothermal Energy*, American Society for Testing and Materials, Philadelphia, 1989 (Annual Book of ASTM Standards, Vol. 12.02).

*Available for purchase from National Technical Information Service, Springfield, VA 22161.

†Available in public technical libraries.

9. In-Service Aged Material Evaluations

F. M. Haggag, S. K. Iskander, R. K. Nanstad, and P. Arakawa

The overall objective of this task is to assess the service-induced degradation of fracture resistance through examination of components exposed during in-nuclear-plant operation. The initial focus of this task is to augment the existing hot-cell testing capability available to the HSSI Program with remote machining capabilities for the fabrication of specimens from samples of activated steel obtained from service-exposed components. During this reporting period, one bid was received from Emco Maier, Inc. (Columbus, Ohio) for a Model VMC-100 machine. This bid was in response to ORNL's specification for purchasing a computer numerically controlled (CNC) vertical milling machine. This machine will be installed inside a hot cell for machining various specimens from components of decommissioned nuclear power plants. Witnessing (by Arakawa and Haggag) of the machining of three specimens [0.5T C(T) compact fracture toughness, CVN, and a miniature flat tensile] was included in the official request for a bid as the acceptance testing (required before delivering the CNC machine to ORNL). The bid from Emco Maier was reviewed and accepted, and a subcontract was placed to purchase the CNC machine, the holding fixture used in machining of the above three specimen geometries, and the software used in acceptance machining. A hard block of steel with special heat treatment to simulate irradiated materials (A 533 with HRC = 30) was sent to Emco Maier for use in machining the three specimens. Delivery of the CNC machine is expected in January 1994. Modification of this machine for remote operation inside the hot cell, as well as personnel training, will be completed in 1995.

10. Correlation Monitor Materials

W. R. Corwin

This task has been established with the explicit purpose of ensuring the continued availability of the pedigreed and extremely well-characterized material now required for inclusion in all additional and future surveillance capsules in commercial LWRs. Having recognized that the only remaining materials qualified for use as a correlation monitor in reactor surveillance capsules are the pieces remaining from the early HSST plates 01, 02, and 03, this task will provide for cataloging, archiving, and distributing the material on behalf of the NRC. The initial activity to be performed in this task will be to identify existing materials and records in preparation for establishing a storage, monitoring, and disbursement facility. During this reporting period, the task of moving the material, previously identified and inventoried as correlation monitor material during the previous reporting period, from its current site at the Y-12 Plant to a controlled-access storage location at ORNL was initiated.

11. Special Technical Assistance

R. K. Nanstad

This task has been established to explicitly emphasize and provide performance and financial monitoring of various analytical and experimental investigations conducted to support the NRC in resolving short-term, high-priority regulatory and research issues. The current activities being performed as part of this task include: evaluating Russian research related to thermal annealing and participating in the ASTM round robin on CVN specimen reconstitution.

11.1 Life Extension

The purpose of this subtask is to evaluate Russian research related to thermal annealing of RPVs. This activity is related, of course, to Task 6 on Annealing Effects in LUS Welds and to Task 13 on Technical Assistance for JCCCNRS Working Groups 3 (radiation embrittlement) and 12 (aging). As part of the activities in Task 13, irradiation, thermal annealing, and reirradiation will be performed on Russian steels. Furthermore, Task 12 on JPDR steel examination includes a subtask on thermal annealing. Thus, because of the substantial amount of thermal annealing research and actual in-service thermal annealing of about 11 reactor vessels by the Russians, a continuing evaluation of their experience is under way in this program. Moreover, M. A. Sokolov, a Russian researcher with expertise in thermal annealing, is on sabbatical at ORNL, and interactions with him on this topic are conducted on a continuing basis. As a result of observations made and presented by Russian researchers, the HSSI Program will focus additional attention on reirradiation embrittlement rates following thermal annealing at 343°C (650°F). The Russian researchers have observed that the extent of embrittlement may be less, for a given fluence, after a 343°C anneal than after a 454°C (850°F) anneal. The amount of recovery and reembrittlement is, of course, material dependent, and experimental observations must be combined with microstructural investigations and modeling to understand the mechanisms.

11.2 CVN Reconstitution Round Robin

The HSSI Program is participating in an ASTM Committee E-10 (Subcommittee E10.02) project on reconstitution of CVN specimens. Part of the project involves a round-robin program with the participation of 11 organizations, both foreign and domestic. All the participants will reconstitute broken CVN specimens according to specifications developed by the subcommittee. The ORNL participation involves the testing of all baseline and reconstituted specimens. ORNL is not involved in the reconstitution exercise of the round robin. The two materials chosen for the study are HSST plate 03 and an LUS weld. All baseline specimens were fabricated by another organization and delivered to ORNL. Baseline tests were conducted with the LUS weld at 1.7 and 93.3°C (35 and 200°F) and with HSST plate 03 at -12 and 93.3°C (10 and 200°F). For each striker [ASTM and International Organization for Standardization (ISO)] and each material, 46 and 23 tests were conducted at the lower and higher temperatures, respectively. The results were reported in the previous semiannual. The ASTM round-robin task leader has distributed the broken specimens to participants for reconstitution. Testing of reconstituted specimens will be performed when at least six participants have supplied specimens.

12. Evaluation of Steel from the JPDR Pressure Vessel

W. R. Corwin and M. A. Sokolov

There is a need to validate the results of irradiation effects research by the examination of material taken directly from the wall of a pressure vessel which has been irradiated during normal service. This task has been included with the HSSI Program to provide just such an evaluation on material from the wall of the pressure vessel from the JPDR.

Even though an informal final agreement has been reached with the Japan Atomic Energy Research Institute (JAERI) on the details of collaboration for research on the material from the vessel of the JPDR, there was very slow movement toward the production of a signed, formal agreement. Following inquiries made into the status of the agreement at JAERI, it was learned that the production of the formal agreement is proceeding but very slowly. ORNL was given assurances that this is typical and does not indicate any lack of interest or other show-stopping problems from JAERI's point of view. It is anticipated that the agreement should be finalized during the next reporting period.

In anticipation of the implementation of the JPDR, which will include the need to test subsize impact specimens, efforts were initiated to expedite the details of fabrication and testing of the subsize specimens. A literature review and analysis of subsize Charpy impact specimen designs, procedures, and data was conducted. A test matrix was developed to study the effects of different geometrical parameters on the relationship between subsize and full-size specimens. Materials with a range of Charpy USEs from about 90 to 300 J have been identified and prepared for machining into subsize specimens to enable the comparison.

13. Technical Assistance for JCCCNRS Working Groups 3 and 12

R. K. Nanstad, M. A. Sokolov, and S. K. Iskander

The purpose of this task is to provide technical support for the efforts of the U.S.-Russian JCCCNRS Working Group 3 on radiation embrittlement and Working Group 12 on aging. Specific activities under this task are: (1) supply of materials and preparation of test specimens for collaborative IAR studies to be conducted in Russia; (2) capsule preparation and initiation of irradiation of Russian specimens within the United States; (3) preparation for, and participation in, Working Groups 3 and 12 meetings; and (4) sponsoring of the assignment at ORNL of M. A. Sokolov of the Russian National Research Center-Kurchatov Institute.

13.1 Irradiation Experiments in Host Country

ORNL supplied CVN and tensile specimens of two materials which have been encapsulated and placed into irradiation positions in the Novovoronezh Unit 5 reactor vessel. Additionally, the Russians provided two weld metals from which ORNL machined CVN and tensile specimens for inclusion in HSST capsule 10.06, which will be irradiated to a target fluence of 1×10^{19} neutrons/cm² (>1 MeV). Some of the Russian specimens will be placed in remote parts of the capsule where the irradiation temperature will be close to 270°C, the operating temperature of some VVER-440 reactors.

13.2 Working Group 3 – J-R Curve Round Robin

As part of the Working Group 3 activities, a J-R curve round-robin program was planned to compare results from U.S. and Russian laboratories. The round robin is conducted by ORNL and includes testing of two each 0.5 and 1TC(T) specimens of two materials at 100°C. The materials are A 533, grade B, class 1 plate (HSST plate 13) and 15Kh2MFA forging. The participating laboratories are ORNL and the U.S. Naval Academy (USNA) for the United States and Kurchatov and Prometey Institutes for Russia. Specimen blanks of HSST plate 13 were machined by ORNL and sent to Russia for machining of specimens. Twelve blanks were also machined into specimens at ORNL, and six were sent to the USNA for testing. The Kurchatov Institute is responsible for supplying specimen blanks of the 15Kh2MFA forging to participants. It is anticipated that some preliminary results may be available to report at the upcoming JCCCNRS Working Group 3 meetings in Washington, D.C., this October 1994.

13.3 Personnel Interactions

The HSSI Program is sponsoring the sabbatical of Dr. Mikhail A. Sokolov at ORNL. Dr. Sokolov arrived at ORNL at the end of December 1992 and is expected to spend 1 year working at ORNL with staff members on the HSSI Program. He will specifically concentrate on activities in the area of thermal annealing of irradiated steels and the use of subsize Charpy impact specimens for irradiated studies. The results of his research will be presented within the particular technical tasks (for example, Task 6 on thermal annealing).

14. Additional Requirements for Materials

D. J. Alexander and R. K. Nanstad

The purpose of this task is to provide technical expertise and assistance in the review of national codes and standards that may be referenced in NRC regulations or guides related to nuclear reactor components. The specific activities to be performed include (1) review of new materials and requirements proposed for inclusion into national codes and standards, of ASME code cases, and of potential deficiencies in proposed supporting technology and data; (2) continuation of long-term aging of stainless steel welds; and (3) continuation of the low-temperature postweld heat treatment study of low-alloy steels.

Present activity is focused on the stainless steel weld aging task. Three stainless steel welds with ferrite contents of either 4, 8, or 12% have been aged at 343°C (650°F) for 3000, 10,000, and 20,000 h. Tensile and CVN specimens have been tested to determine the effect of such aging on the mechanical properties of the material, while optical metallography, scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atom-probe field-ion microscopy (APFIM) have been used to follow the changes in the microstructure.

Aging has little effect on the tensile properties at room temperature for any of the materials. However, the impact properties are noticeably affected. There is little change in the behavior of the 4% ferrite material, but the materials that contain 8 or 12% ferrite do respond to aging. As the aging time increases, the ductile-to-brittle transition temperature increases, and the upper-shelf energy level decreases. The magnitude of the changes increase with an increasing ferrite content.

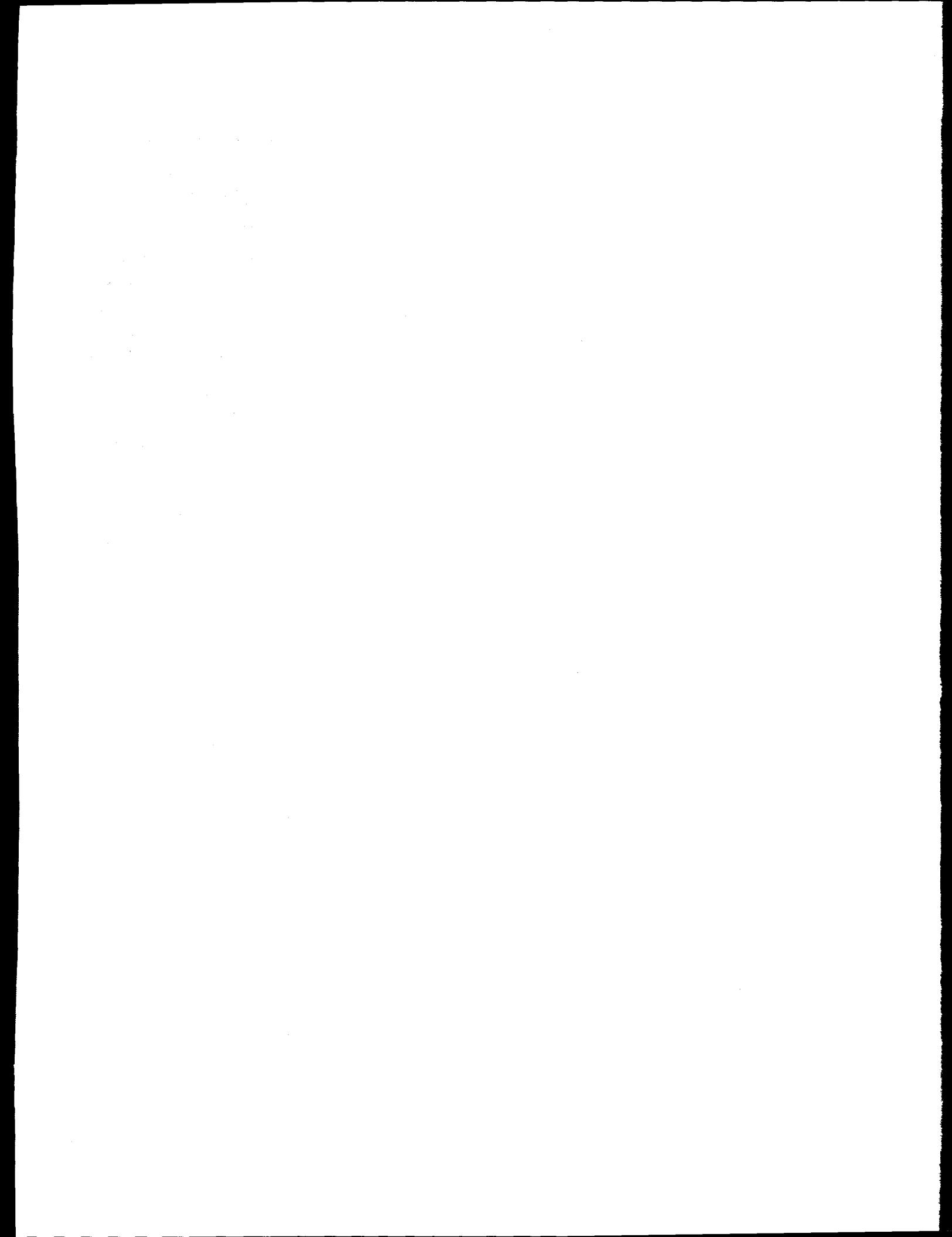
The TEM and APFIM examinations have shown that a spinodal decomposition reaction occurs in the ferrite, with iron-rich and chromium-enriched regions forming early in the aging process. This results in significant hardening of the ferrite. SEM examinations show that the fracture surfaces change from ductile, dimpled surfaces to ones containing large, flat regions that display what appear to be cleavage markings. It is believed that the spinodal decomposition hardens the ferrite; in the transition temperature regime, this favors a change in the fracture mode of the ferrite from ductile microvoid coalescence to a low-energy cleavage mode of fracture.

Additional material from each of these weldments is being aged to reach 50,000 h, which will be achieved in February 1994. This material will then be tested to determine if further degradation of the impact properties results from the longer aging time. A NUREG report is being written to summarize the results from the mechanical testing and the microstructural examinations to date.

CONVERSION FACTORS*

SI Unit	English unit	Factor
mm	in.	0.0393701
cm	in.	0.393701
m	ft	3.28084
m/s	ft/s	3.28084
kN	lb _f	224.809
kPa	psi	0.145038
MPa	ksi	0.145038
MPa [•] √m	ksi [•] √in.	0.910048
J	ft [•] lb	0.737562
K	°F or °R	1.8
kJ/m ²	in. [•] lb/in. ²	5.71015
W [•] m ⁻³ [•] K ⁻¹	Btu/h [•] ft ² [•] °F	1.176110
kg	lb	2.20462
kg/m ³	lb/in. ³	3.61273 [•] 10 ⁻⁵
mm/N	in./lb _f	0.175127
$T(^{\circ}\text{F}) = 1.8(^{\circ}\text{C}) + 32$		

*Multiply SI quantity by given factor to obtain English quantity.



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<p>The goal of the Heavy-Section Steel Irradiation Program is to provide a thorough, quantitative assessment of effects of neutron irradiation on material behavior, and in particular the fracture toughness properties, of typical pressure vessel steels as they relate to light-water reactor pressure-vessel integrity. Effects of specimen size, material chemistry, product form and microstructure, irradiation fluence, flux, temperature and spectrum, and post-irradiation annealing are being examined on a wide range of fracture properties. The HSSI Program is arranged into 14 tasks: (1) program management, (2) fracture toughness (K_{Ic}) curve shift in high-copper welds, (3) crack-arrest toughness (K_{Ia}) curve shift in high-copper welds, (4) irradiation effects on cladding, (5) K_{Ic} and K_{Ia} curve shifts in low upper-shelf welds, (6) annealing effects in low upper-shelf welds, (7) irradiation effects in a commercial low upper-shelf weld, (8) microstructural analysis of irradiation effects, (9) in-service aged material evaluations, (10) correlation monitor materials, (11) special technical assistance, (12) JPDR steel examination, (13) technical assistance for JCCCNRS Working Groups 3 and 12, and (14) additional requirements for materials. This report provides an overview of the activities within each of these tasks from April to September 1993.</p>			
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