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# Heavy-Section Steel Irradiation Program

Semiannual Progress Report for  
September 1993 – March 1994

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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 that have the potential for major contamination release. The RPV is the only component in the primary pressure boundary for which, if it should rupture, the engineering safety systems cannot assure protection from core damage. 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 that occurs during service.

For this reason, the Heavy-Section Steel Irradiation (HSSI) Program has been established; its primary goal 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 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. At the beginning of this reporting period, the HSSI Program was arranged into 14 tasks. Halfway through the period, the program was reorganized into 13 somewhat different tasks with the acceptance of the 189 for calendar year 1994. Work from all of the prior tasks was continued in the program, although the tasks were renumbered and in some cases combined where appropriate. In addition, two new tasks were initiated: one on irradiation effects in old-practice plate and heat-affected zone materials and one on the integration of research results into the codes and standards that pertain to the methods used to shift fracture properties to account for irradiation embrittlement. To maintain continuity, the task-numbering system in place at the beginning of the reporting period is used throughout this report: (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 Working Groups 3 and 12, and (14) additional requirements for materials. The new task numbers will be used in future semiannual progress reports. Also, because work in the two new tasks has just begun, reporting of it will be deferred until the next progress report.

During this period, the report on the duplex-type crack-arrest specimen tests from Phase II of the  $K_{Ia}$  program was issued, and final preparations for testing the large, irradiated crack-arrest specimens from the Italian Committee for Research and Development of Nuclear Energy and Alternative Energies were completed. Tests on undersize Charpy V-notch (CVN) energy specimens in the irradiated weld 73W, annealed at 454°C (850°F) and 343°C (650°F) for 1 to 14 days, were completed. While CVN 41-J (30 ft-lb) transition temperature almost fully recovered for the longest period studied at the higher temperature, recovery was to lesser degrees for the shorter periods. No significant recovery of these CVN properties was observed for the lower temperature. The effects of annealing on recovery of the J-R curve were also studied on six LUS welds. The annealing of the six irradiated welds at 454°C (850°F) for 168 h resulted in full recovery of  $J_{Ic}$  and tearing modulus. The NUREG report, *Unirradiated Material Properties of Midland Weld WF-70*, was issued. Fracture toughness tests of the unirradiated material show that the welds in the nozzle course and bellline regions differ in their fracture toughness transition temperatures (measured at a  $K_J$  value of 100 MPa $\sqrt{m}$ ). The necessary calculations were completed to permit an evaluation of the relative importance of copper-rich precipitates and point defect clusters in RPV embrittlement. The results are described in detail in a draft NUREG report in preparation. In addition, the Oak Ridge National Laboratory (ORNL) investigation of the embrittlement of the High Flux Isotope Reactor pressure vessel indicated that an unusually large ratio of the high-energy gamma-ray flux to fast-neutron flux is most likely responsible for the apparently accelerated embrittlement.

Final plans were made, and a section of HSSI weld 72W was sent to AEA Technology, Harwell, for use in testing of notched and precracked round bars to ascertain the correspondence of their data to that for more traditional

fracture specimens. A total of ten materials/conditions have been selected, specimens have been fabricated, and testing has begun for the study of subsized impact specimens. Five designs of subsized specimens were chosen for this study, the results of which will be compared with those from previously tested full-size specimens. For the American Society for Testing and Materials reconstituted Charpy impact specimen project, reconstituted specimens have been received from six of the participants. This group of specimens will be tested in the next 2 months, and specimens received subsequently will be tested as a second group. The final JPDR collaborative research agreement was signed by Japan Atomic Energy Research Institute (JAERI) and sent to ORNL for approval. JAERI shipped 16 full-thickness trepanns, each approximately 87 mm in diameter, of material from the wall of the JPDR to ORNL, where it will be examined. The material received at ORNL consists of four types: weld metal and base metal, each in both the irradiated and in nominally, thermally aged only conditions. Specimens of two Russian welds were fabricated and placed in HSSI capsule 10.06 for irradiation in the University of Michigan Ford Nuclear Reactor.

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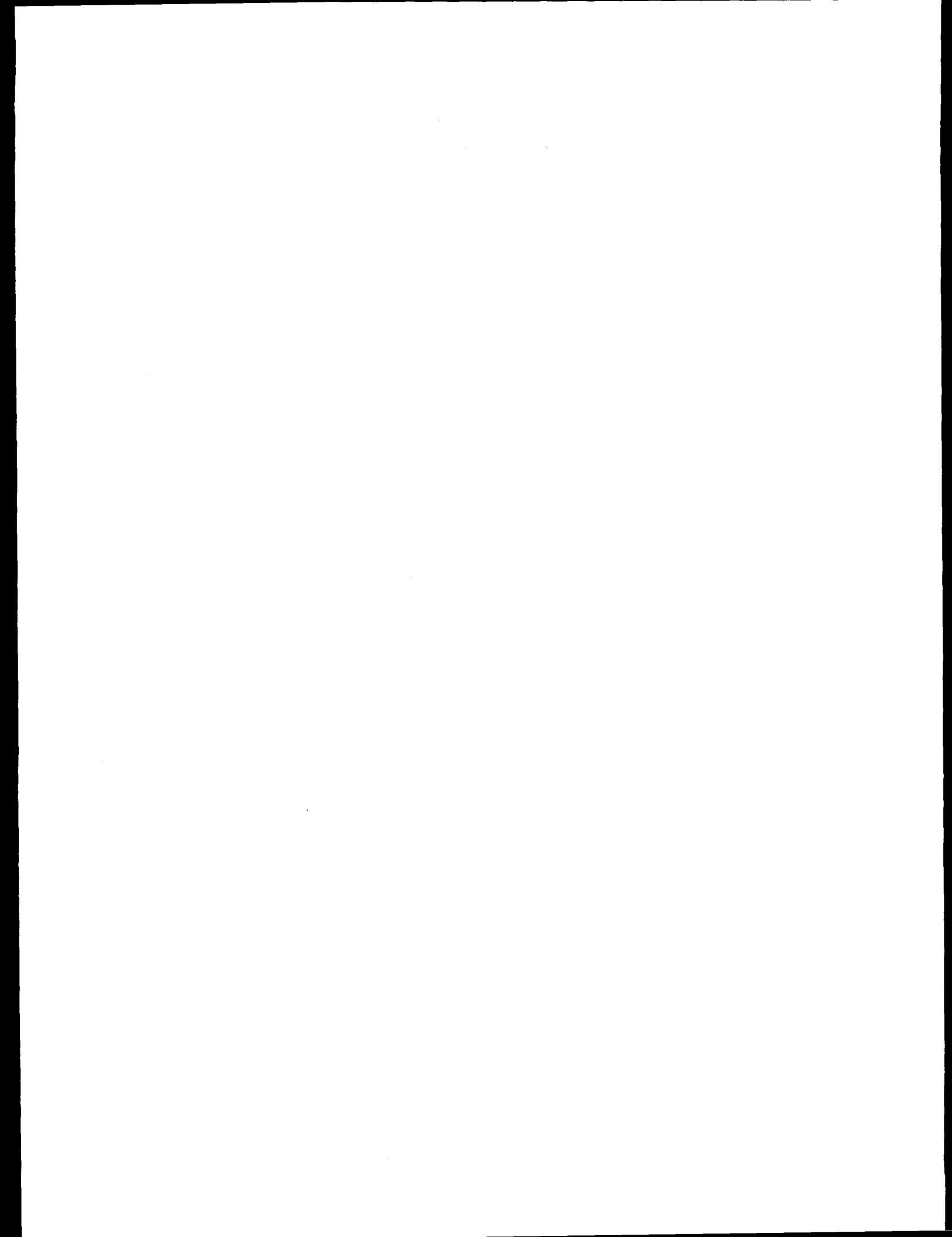
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## Preface

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 that 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 October 1993 to March 1994. 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:

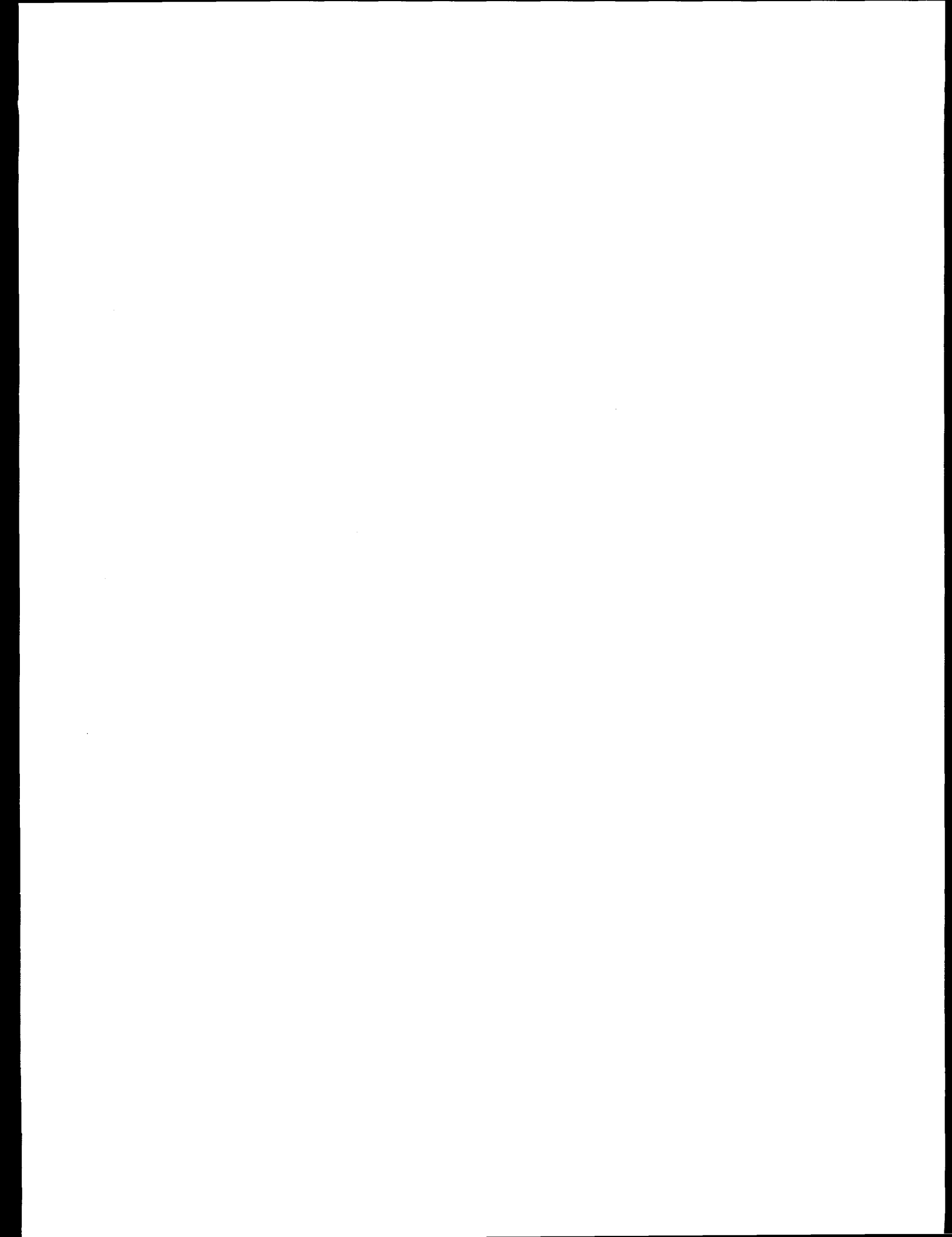
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(ORNL/TM-11568/V1&N1)  
NUREG/CR-5591, Vol. 1, No. 2  
(ORNL/TM-11568/V1&N2)  
NUREG/CR-5591, Vol. 2, No. 1  
(ORNL/TM-11568/V2&N1)  
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(ORNL/TM-11568/V2&N2)  
NUREG/CR-5591, Vol. 3  
(ORNL/TM-11568/V3)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

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

ORNL-4918  
ORNL-4971  
ORNL/TM-4655 (Vol. II)  
ORNL/TM-4729 (Vol. II)  
ORNL/TM-4805 (Vol. II)  
ORNL/TM-4914 (Vol. II)  
ORNL/TM-5021 (Vol. II)  
ORNL/TM-5170  
ORNL/NUREG/TM-3  
ORNL/NUREG/TM-28  
ORNL/NUREG/TM-49  
ORNL/NUREG/TM-64  
ORNL/NUREG/TM-94  
ORNL/NUREG/TM-120  
ORNL/NUREG/TM-147  
ORNL/NUREG/TM-166  
ORNL/NUREG/TM-194  
ORNL/NUREG/TM-209  
ORNL/NUREG/TM-239  
NUREG/CR-0476 (ORNL/NUREG/TM-275)  
NUREG/CR-0656 (ORNL/NUREG/TM-298)  
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NUREG/CR-1477 (ORNL/NUREG/TM-393)  
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NUREG/CR-3334, Vol. 2 (ORNL/TM-8787/V2)  
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NUREG/CR-4219, Vol. 2 (ORNL/TM-9593/V2)  
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# Summary

## 1. Program Management

At the beginning of this reporting period, the Heavy Section Steel Irradiation (HSSI) Program was arranged into 14 tasks. Halfway through the period, the program was reorganized into 13 somewhat different tasks with the acceptance of the 189 for calendar year 1994. Work from all of the prior tasks was continued in the program, although the tasks were renumbered and in some cases combined where appropriate. In addition, two new tasks were initiated: one on irradiation effects in old-practice plate and heat-affected zone materials and one on the integration of research results into the codes and standards that pertain to the methods used to shift fracture properties to account for irradiation embrittlement. To maintain continuity, the task-numbering system in place at the beginning of the reporting period is used throughout this report: (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 (JDPR) 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. The new task numbers will be used in future semiannual progress reports. Also, because work in the two new tasks has just begun, reporting of it will be deferred until the next progress report. During the report period, 17 technical presentations were given, and 8 technical papers were published.

## 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, 0.23 and 0.31 wt %, submerged-arc welds (72W and 73W respectively). Phase I of this task was completed and reported in NUREG/CR-5913. The objective of Phase II is to obtain postirradiation fracture toughness data to a neutron fluence of  $5 \times 10^{19}$  neutrons/cm<sup>2</sup> (>1 MeV). Preparations are under way for machining test specimens for both welds. Included as a part of this task is the development of an American Society for Testing and Materials (ASTM) standard practice for testing in the ductile/brittle transition range. The fifth version of this draft standard has been distributed to interested parties within ASTM for comment.

## 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 submerged-arc welds. 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 previously 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. A report giving the results of testing of the HSSI duplex crack-arrest specimens was published (NUREG/CR-6139). The task also includes the testing of nine irradiated Italian crack-arrest specimens of pressure vessel steel from the Italian Committee for Research and Development of Nuclear Energy and Alternative Energies. Preparations for moving the Italian specimens into the hot cell have been completed. Efforts are under way to design and to fabricate a special crack-mouth opening displacement gage for use with the Italian specimens.

## 4. Irradiation Effects on 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. Progress on this task during this reporting period is discussed in Chapter 9.

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

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 two welds with high-copper and low CVN upper-shelf energy. The information developed under this task augments that obtained in a similar irradiation experiment performed on two welds with high CVN upper-shelf energy under the HSSI Fifth and Sixth Irradiation Series. The planning phase of this task resulted in the decision to fabricate three trial welds using Linde 80 welding flux. Initial contacts have been made with a vendor, and a purchase requisition for these welds will be executed in the next reporting period.

## 6. Annealing Effects in LUS 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. Tests on undersize CVN energy specimens in the irradiated and irradiated/annealed conditions were completed. The specimens were fabricated from the well-characterized high-copper submerged-arc weld 73W used for the Fifth and Sixth Irradiation Series. The CVN specimens were annealed at 454°C (850°F) between 1 and 14 days. While the CVN 41-J (30 ft-lb) transition temperature almost fully recovered for the longest period studied, recovery was to lesser degrees for the shorter periods. No significant recovery of these CVN properties was observed for a 7-day anneal at 343°C (650°F). At 454°C (850°F) and the durations studied, the values of the upper-shelf impact energy of irradiated and annealed weld metal exceeded that in the unirradiated condition. A similar behavior was observed for aging the unirradiated weld at 454°C (850°F) for 1 week.

The effects of annealing on recovery of the J-R curve were also studied on six LUS welds fabricated with materials and procedures used in early pressurized-water reactor pressure vessels (RPVs) that remain from the Second, Third, and Fourth Irradiation Series. Previous studies of these six welds showed that irradiation decreased the values of  $J_{Ic}$  and tearing modulus (T) significantly from the unirradiated values, where  $J_{Ic}$  is a critical crack growth initiation toughness value. The annealing of the six irradiated welds at 454°C (850°F) for 168 h resulted in full recovery of  $J_{Ic}$ . The same recovery was observed in the values of T. Thus, annealing of the welds studied at 454°C (850°F) showed that the properties degraded by neutron irradiation were substantially recovered. In many cases the fracture toughness, upper-shelf energy, and  $J_{Ic}$  of irradiated and annealed welds are greater than in the unirradiated condition.

A special fixture for measuring the lateral expansion of CVN specimens has been designed, fabricated, calibrated, and installed in the hot cell. The design of the new irradiation, annealing, and reirradiation capsule and facility is progressing. The first two capsules will be installed on the east face of the University of Michigan Ford Nuclear Reactor. The data acquisition and control system will also be upgraded so it can control all the capsules being irradiated. Preliminary tests at 150°C (302°F) of the recently purchased regulated-atmosphere annealing furnace have been completed. This furnace may be used for future annealing work in the hot cell.

## 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 submerged-arc weld 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, a submerged-arc weld 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, fracture toughness tests of the unirradiated material have been completed, analyzed, and presented in a NUREG report. The results show that the welds in the nozzle course and beltline regions differ in their fracture toughness transition temperatures (measured at a  $K_J$  value of 100 MPa $\sqrt{m}$ ).

## **8. Microstructural Analysis of Radiation Effects**

The overall long-term goal of this task is to develop a physically based model that 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 necessary calculations were completed to permit an evaluation of the relative importance of copper-rich precipitates and point defect clusters in RPV embrittlement. The results are described in detail in a draft NUREG report. In addition, the Oak Ridge National Laboratory (ORNL) investigation of the embrittlement of the High Flux Isotope Reactor pressure vessel indicated that an unusually large ratio of the high-energy gamma-ray flux to fast neutron flux is most likely responsible for the apparently accelerated embrittlement. Gamma rays can indirectly cause atomic displacements by first generating high-energy electrons by either pair production or Compton scattering. When these electrons have energies above a few hundred kiloelectron volts, they can displace lattice atoms. Because the fluxes of high-energy neutrons and gamma rays are comparable and because the displacement cross section for these electron events is about 1/1000 of that for fast neutrons, such displacements are usually insignificant.

## **9. In-Service Irradiated and Aged Material Evaluations**

The principal 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. This task has been modified to include other various subtasks with the objectives to evaluate material properties in components of nuclear reactors, including the effects of aging and irradiation. For this progress report, the information reported here includes work from Task 4, Irradiation Effects on Cladding, and Task 14, Additional Requirements for Materials. During this reporting period, procurement of the computer numerically controlled machining center was well under way following verification of the capabilities of the specific machine being procured; the machine is expected to be delivered in the summer of 1994. Testing of some thermally aged type 308 stainless steel welds and type 308 stainless steel weld overlay cladding specimens was completed, and aging to longer times is being continued.

## **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. 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 (HSST) Plates 01, 02, and 03; thus, this task will provide for cataloging, archiving, and distributing the material on behalf of the Nuclear Regulatory Commission (NRC). During this reporting period, the preparations for moving the material to a controlled-access storage location at ORNL were continued, detailed planning for the

controlled-access location for storage was initiated, and correlation monitor material from HSST Plate 02 was shipped to A. Kumar at the University of Missouri, Rolla, for his use in studies of irradiation assessment using subsize CVN specimens.

## **11. Special Technical Assistance**

This task has been included with the HSSI Program to provide a vehicle in which to conduct and to monitor short-term, high-priority subtasks and 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. During this reporting period, a section of HSSI weld 72W was sent to AEA Technology, Harwell, for use in testing of notched and precracked round bars. For the subsize Charpy impact project, a total of ten materials/conditions have been selected, and specimens have been fabricated. Five designs of subsize specimens were chosen for this study, and the results will be compared with those from previously tested full-size specimens. Tests of all the subsize specimens from HSST Plate 02 have been completed, and testing of the other materials is in progress. For the reconstituted Charpy impact specimen project, reconstituted specimens have been received from six of the participants. This group of specimens will be tested early in the next reporting period, and specimens received subsequently will be tested as a second group.

## **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 that 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, the final agreement was signed by the Japan Atomic Energy Research Institute (JAERI) and sent to ORNL for approval. The agreement should be finalized during the next reporting period. JAERI shipped the irradiated material from the wall of the JPDR that will be examined at ORNL. On its arrival at ORNL, arrangements were made to move it into the hot cells where it is to be machined and examined. The material on its arrival at ORNL consists of 16 full-thickness trepans, each approximately 87 mm in diameter, containing four types of material: weld metal and base metal, each in both the irradiated condition (from the bellline) and in, nominally, the thermally aged only condition (from the upper flange).

In anticipation of the implementation of the JPDR testing program, that will include the need to test subsize impact specimens, approximately 70% of the testing of a matrix that was developed to study the effects of different geometrical parameters on the relationship between subsize and full-size impact specimens was completed. This matrix includes five types of subsize specimens and ten materials with a wide range of Charpy transition temperatures and upper-shelf energies.

## **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 irradiation, annealing, and reirradiation 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. Specimens of two Russian welds were fabricated and placed in HSSI capsule 10.06 for irradiation in the

University of Michigan Ford Nuclear Reactor. The irradiation should be completed at the end of FY 1994. Meetings of Working Group 3 were held in Rockville and Annapolis, Maryland, in October 1993; six participants were from the ORNL HSSI Program.

#### **14. Additional Requirements for Materials**

The purpose of this task is to provide materials 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. Progress on this task during this reporting period is discussed in Chapters 9 and 11.

# Heavy-Section Steel Irradiation Program Semiannual Progress Report for September 1993 through March 1994<sup>\*†</sup>

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 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 nil-ductility transition, 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 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 light-water reactor 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.

At the beginning of this reporting period, the HSSI Program was arranged into 14 tasks. Halfway through the period, the program was reorganized into 13 somewhat different tasks with the acceptance of the 189 for calendar year 1994. Work from all of the prior tasks was continued in the program, although the tasks were renumbered and

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in some cases combined where appropriate. In addition, two new tasks were initiated: one on irradiation effects in old-practice plate and heat-affected zone materials and one on the explicit integration of research results into the codes and standards that pertain to the methods used to shift fracture properties to account for irradiation embrittlement. To maintain continuity, the task-numbering system in place at the beginning of the reporting period is used throughout this report: (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 Development Reactor (JPDR) 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, seven program briefings, reviews, or presentations were made by the HSSI staff during program reviews and visits with NRC staff or others. Eight technical papers were published.<sup>1-8</sup> In addition, 10 technical presentations were made.<sup>9-18</sup>

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<sup>†</sup>Available in public technical libraries.

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

R. K. Nanstad and D. E. McCabe

### 2.0 Introduction

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 (72W and 73W respectively). All planned unirradiated and irradiated testing for Phase 1 of the Fifth Irradiation Series has been completed. The results of all testing and analyses have been reported. Included in this task is a continuing evaluation of material behavior in the transition temperature region. This includes evaluation of the use of small specimens that exhibit elastic-plastic fracture behavior to represent the fracture toughness of larger specimens. A major part of this effort is the development of a standard practice for testing in the transition temperature region; progress in this area is described in this chapter.

### 2.1 Phase II of the Fifth Irradiation Series

The objective of Phase II of this series is to obtain postirradiation fracture toughness data to a neutron fluence of  $5 \times 10^{19}$  neutrons/cm<sup>2</sup> (>1 MeV). Archive material is available, and preparations are under way for machining test specimens for welds 72W and 73W. Fracture toughness, 1 TC(T), CVN, and tensile specimens will be irradiated at 288°C (550°F). The detailed specimen matrix will be dependent on final design of the irradiation facility and capsules currently under way.

### 2.2 Development of ASTM Standard Practice in Transition Range

The fifth draft of the proposed American Society for Testing and Materials (ASTM) test practice, Development of Test Practice (Method) for Fracture Toughness in the Transition Range, has been improved in several ways over the past period. As an example, the procedure to be followed in making a best fit to the experimental data, when those data are plotted in Weibull coordinates, was enormously simplified. Another upgrade was the further clarification of what constitutes an invalid  $K_{Jc}$  datum. Data become invalid when (1) no fracture occurs prior to termination of specimen loading, (2) toughness at  $K_{Jc}$  instability is so high as to violate a controlled constraint condition, where  $\beta_c > 0.5 \pi$ , and (3) slow-stable crack growth at  $K_{Jc}$  instability exceeds 5% of the initial remaining ligament dimension. Another modification added to the proposed practice was to insert a statistical data-censoring practice to be used when data sets contain one or more invalid datum. If one is prepared to accept increased penalty in the form of enlarged estimates in standard deviations, it is possible to deal with smaller groups of  $K_{Jc}$  data. This possibility is exploratory work currently under way. Another avenue of exploration is to develop new ideas for transforming single  $K_{Jc}$  datum into  $K_{Ic}$  valid datum.

There have been two revisions to the proposed practice in the period covered by this report. The aim is to have all work completed by the fall meetings of the sponsoring Task Group E08.08.03 in ASTM Committee E-08 on Fatigue and Fracture.

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

S. K. Iskander, E. T. Manneschildt, and K. W. Boling

### 3.0 Introduction

The objective of this task is to develop data addressing the current method of shifting the ASTM fracture toughness crack-arrest toughness ( $K_{Ia}$ ) curve to account for irradiation embrittlement in high-copper welds. The activities performed in this reporting period included preparations for testing of the irradiated Italian crack-arrest specimens. The report giving the results of the duplex crack-arrest specimen testing has been published.<sup>1</sup>

### 3.1 Preparations for Testing Irradiated Crack-Arrest Specimens Supplied by ENEA

As described in the previous semiannual report, the new remote crack-arrest fixture was built and successfully tested using unirradiated specimens from Midland weld. It will be used to test the large irradiated crack-arrest specimens from the Italian Committee for Research and Development of Nuclear Energy and Alternative Energies (ENEA). The results will have usefulness and applicability to the safety assessment of U.S. reactor pressure vessels. A method to measure the crack-mouth opening of the ENEA crack-arrest specimens must still be devised. Some background information on the ENEA program has been given in a previous semiannual report.

The irradiated Italian crack-arrest specimens were moved to the hot-cell area in preparation for their testing in the next period. Because of their size, the three large specimens will be particularly difficult to move into the cells. The usual procedure of using a bottom-loading cask and lowering the specimens into the cell through a roof opening cannot be used because casks sufficiently large to hold the specimens are too heavy for the available crane and exceed the allowable load for the roof of the hot cell. The large specimens will be introduced through a cell back door and then moved using manipulators through the adjacent cells to the cell in which they will be tested. Incidentally, the weight of the specimens is very close to the lifting capacity of the manipulators. The six smaller specimens do not present any problems to move into the cells.

Before the specimens can be tested, clip-gage blocks for the conical-ended clip gage used for measuring the crack-mouth opening displacement (CMOD) must be designed and fabricated. This type of clip gage has been previously used successfully at ORNL to test irradiated crack-arrest specimens.<sup>2</sup> The CMOD will be measured at a distance from the loading line greater than the 0.25 W distance prescribed in the ASTM Test for Determining Plane-Strain Crack-Arrest Fracture Toughness,  $K_{Ia}$ , of Ferritic Steels (E 1221). The CMOD will be slightly larger than if it was measured at 0.25 W and will be adjusted accordingly. The increase in CMOD due to the increased measuring distance is estimated to be less than 5%.

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

## **4. Irradiation Effects on Cladding**

**F. M. Haggag**

Progress on this task during this reporting period is discussed in Chapter 9.

## 5. $K_{Ic}$ and $K_{Ia}$ Curve Shifts in Low Upper-Shelf Welds

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

### 5.0 Introduction

This task examines the fracture toughness curve shifts and changes in shape for irradiated welds with Charpy LUS energy. The information developed under this task augments that obtained in a similar irradiation experiment performed on two Charpy high upper-shelf energy weldments under the Fifth and Sixth Irradiation Series. The results will provide an expanded basis for accounting for irradiation-induced embrittlement in RPV materials. After discussions with various parties, it was decided to fabricate three trial welds using Linde 80 flux. The three welds to be fabricated are to include one weld using 73W wire; the remaining two contain 0.31 and 0.45% copper and impurities that are typical of older generation nuclear pressure vessels. Initial contacts were made with the vendor that fabricated the welds for the Fifth and Sixth Series to obtain approximate costs and schedules for the fabrication of the welds. The purchase requisition for these welds will be made in the next period.

### 5.1 Discussion of Reasons for Decision to Purchase the Three Development Welds

Each weld will be approximately 380 mm long (15 in.) and will be fabricated using a 1140-mm (45-in.) segment of the A 508 class 2 base metal remaining from the Midland RPV and Linde 80 flux that will result in LUS welds. The welds are to be trial welds, and if their evaluation is successful, sufficient quantities are to be fabricated for the Eighth and Ninth Irradiation Series. The objectives of the Eighth Series are similar to those of the Fifth and Sixth Series (i.e., to investigate the effect of irradiation on LUS welds on  $K_{Ic}$  and  $K_{Ia}$  curve shifts and shapes). The objective of the Ninth Series is to investigate the irradiation, annealing, and reirradiation behavior of critical materials.

After discussions with various parties, it was decided to fabricate three trial welds using Linde 80 flux. The three welds to be fabricated are to include one weld using 73W wire; the remaining two contain 0.31 and 0.45% copper and impurities that are typical of older generation nuclear pressure vessels. For purposes of this discussion, they are designated welds A, B, and C. Weld A will be fabricated using the 73W weld wire that was used in the HSSI Fifth and Sixth Irradiation Series and Linde 80 flux. The chemical composition of both wire and resulting weld using Linde 124 flux is given in Table 1. The purpose of weld A is twofold: First, the distribution of copper throughout the new LUS weld A will be checked for uniformity using electron microprobe analysis and compared with those of welds B and C (being prepared at the same time) as well as with the copper distribution of HSSI weld 73W. Secondly, the effect of Linde 80 flux on the resulting weld's chemical composition must be determined. This weld could be a candidate weld for the Eighth and Ninth Irradiation Series.

Welds B and C will be prepared using the ABB-CE process of adding copper during welding. There are also two reasons for preparing welds B and C: (1) to change the copper content from that used in the Fifth and Sixth Irradiation Series and (2) to accommodate a potential lack of sufficient quantities of 73W weld wire to meet all the requirements of the Eighth and Ninth Irradiation Series.

Welds B and C will also be prepared using the same base metal and flux, but the weld wire will be purchased by ABB-CE. They will try to obtain a wire that was used in earlier vintage RPVs to ensure that the amount of phosphorous, sulfur, etc., will be higher than that of the weld 73W (i.e., representative of the early vintage LUS welds currently in service). Most of the elements will be controlled by the available weld wire, although some, notably silicon, will be picked up from the flux. This wire will be purchased from a commercial vendor using an electrode specification from ASME SFA-5.23. For example, the range of the chemical compositions of the LUS welds investigated in the Second and Third HSSI Series, typical of early vintage, high-copper, LUS welds, is shown in Table 2. The content of bulk copper is crucial and is something the vendor can control.

**Table 1. Chemical compositions of weld wire and submerged-arc weld 73W  
(final welds were fabricated using Linde 124 flux, lot 0103)**

Element	Chemical analysis, drum 2 <sup>a</sup> (wt %)	Final welds <sup>b</sup>	
		Mean (wt %)	Standard deviation (wt %)
Carbon	0.12	0.098	0.007
Manganese	2.04	1.56	0.026
Phosphorus	0.003	0.005	0.0004
Sulfur	0.002	0.005	0.0006
Silicon	0.07	0.45	0.027
Nickel	0.62	0.60	0.006
Chromium	0.27	0.25	0.006
Molybdenum	0.55	0.58	0.009
Vanadium	0.003	0.003	0.0001
Columbium/Tantalum	<0.01		
Titanium	<0.01		
Cobalt	0.032	0.030	
<b>Copper</b>	<b>0.35</b>	<b>0.31</b>	<b>0.010</b>
Aluminum	0.006	0.006	
Boron	<0.001		
Tungsten	<0.01		
Arsenic	0.003	0.002	
Tin	0.002	0.003	
Zirconium	<0.001		

<sup>a</sup>Check Analysis of HSST-73, Heat 87986 Drum Number 2, Filler Welding Wire Composition, wt %.  
Source: *Test Welds Using HSST72 and 73 Filler Wire - for Martin Marietta Energy Systems, Inc., Purchase Specification ORNL-RB2111111, Subcontract 76X-6700V*, prepared by C. D. Hackett and C. T. Ward, Final Report, Feb. 1, 1985, MML-85-24, C-E Contract 6083, Project No. 900581, Shop Order 98130390.

<sup>b</sup>R. K. Nanstad, F. M. Haggag, D. E. McCabe, S. K. Iskander, K. O. Bowman, and B. H. Menke, *Irradiation Effects on Fracture Toughness of Two High-Copper Submerged-Arc Welds, HSSI Series 5, NUREG/CR-5913, Vol. 1 (ORNL/TM-12156/V1)*, August 1992.

**Table 2. Average, minimum, and maximum values of chemical compositions of HSSI welds 61W through 67W**

(Average is the average of all welds, whereas the minimum and maximum values do not necessarily correspond simultaneously to any one particular weld, but the values observed for the element in all the welds)

Weld	Composition (wt %)									
	C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V
Average	0.086	1.530	0.016	0.011	0.557	0.110	0.595	0.391	0.268	0.008
Minimum	0.070	1.400	0.010	0.007	0.410	0.067	0.480	0.360	0.140	0.005
Maximum	0.110	1.670	0.021	0.017	0.670	0.180	0.702	0.440	0.490	0.012

Arguments for the various target bulk copper compositions include:

1. A copper content of 0.31%. The primary advantage is the same copper content as HSSI weld 73W. Useful comparisons may be easier to make between the new weld and the available large data base of the HSSI weld 73W.
2. A copper content of 0.45%. A copper level as high as 0.49% has been observed in the Midland nozzle welds. Because both beltline and nozzle welds were Babcock and Wilcox weld WF-70, such a high-copper content could have ended up in the beltline also. Assuming that flaws are initiated by the weakest "link," useful *bounding* information may be obtained on the effect of such a high-copper content on degradation of toughness due to irradiation. The highest content in NRC *Regulatory Guide 1.99, Rev. 2*, for welds is 0.40%. The guide is based on surveillance data with very sparse data from welds with such high-copper contents. This weld could provide additional data. Another advantage of such a high-copper content is its potentially increased radiation sensitivity, allowing shorter radiation times to achieve the changes that could be unambiguously attributed to neutron damage rather than scatter.
3. A copper content of 0.35%. This is, as are the others, a relatively high value and would provide data for welds within the copper content that is assumed in *Regulatory Guide 1.99, Rev. 2* when the copper content is not known.

Because the required linear length of the trial weld is small, it was decided to obtain three welds using Linde 80 flux. Weld A will use HSSI weld wire 73W remaining from the Fifth and Sixth Series. The other two will also use Linde 80 flux but with a commercially obtained weld wire as mentioned above. Welds B and C will have target copper levels of 0.31 and 0.45% respectively.

A purchase requisition will be prepared, and the welds ordered in the next reporting period.

## 6. Annealing Effects in Low Upper-Shelf Welds (Series 9)

S. K. Iskander, M. A. Sokolov, R. K. Nanstad,  
E. T. Manneschildt, and K. W. Boling

### 6.0 Introduction

The purpose of the Ninth Irradiation Series is to evaluate the correlation between fracture toughness and CVN impact energy during irradiation, annealing, and reirradiation. Tests on so-called undersize CVN impact energy specimens in the irradiated and irradiated/annealed conditions were completed. The specimens were fabricated from the well-characterized, high-copper, submerged-arc, HSSI weld 73W used for the Fifth and Sixth Irradiation Series. The CVN specimens were annealed at 454°C (850°F) for lengths of time varying between 1 and 14 days. While the CVN 41-J (30 ft-lb) impact energy level transition temperature almost fully recovered for the longest period studied, recovery was to lesser degrees for the shorter periods. No significant recovery of these CVN properties was observed for a 7-day anneal at 343°C (650°F). At 454°C (850°F) and for the durations studied, the values of the upper-shelf impact energy of irradiated and annealed weld metal exceeded that in the unirradiated condition. A similar behavior was observed for aging the unirradiated weld at 454°C (850°F) for 1 week.

The effects of annealing on recovery of the J-R curve were also studied on six LUS welds fabricated with materials and procedures used in early pressurized-water RPVs. The specimens tested were those remaining from the Second, Third, and Fourth Irradiation Series. Previous studies of the six welds from the Second and Third Series showed that irradiation decreased the values of  $J_{IC}$  and tearing modulus (T) significantly from the unirradiated values. Annealing of the six irradiated welds at 454°C (850°F) for 168 h resulted in full recovery of  $J_{IC}$ . Similar recovery was observed in the values of T for the welds.

A special fixture for measuring the lateral expansion of CVN specimens has been designed, fabricated, calibrated, and installed in the hot cell. The lateral expansion of the recently tested, undersized, irradiated/annealed, 73W CVN specimens has been measured. The lateral expansion of the 44 CVN specimens from 72W and 73E that were irradiated in the Sixth Irradiation Series capsules with the crack-arrest specimens will be measured soon.

### 6.1 Material Used for Annealing Irradiated High-Copper Welds

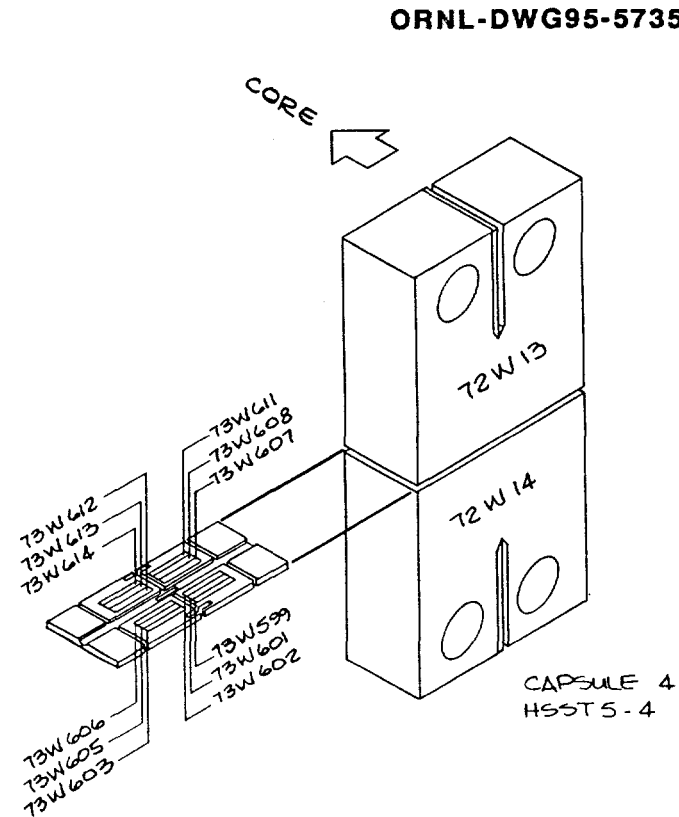
The commercially fabricated submerged-arc weld used for this study, HSSI weld 73W, has been very extensively characterized in other HSSI tasks.<sup>1-5</sup> Large variations in the copper content (e.g., from 0.22 to 0.46%) have been observed in welds fabricated using weld wire with hot-dipped copper coating.<sup>6</sup> To avoid these large variations, the 73W weld wire was fabricated with copper added to the melt; consequently, the variation in copper content is very small. The chemical composition and standard deviation of HSSI weld 73W (obtained from a large number of analyses) are shown in Table 3.

Table 3. Chemical composition and standard deviation of the various elements analyzed in HSSI weld 73W

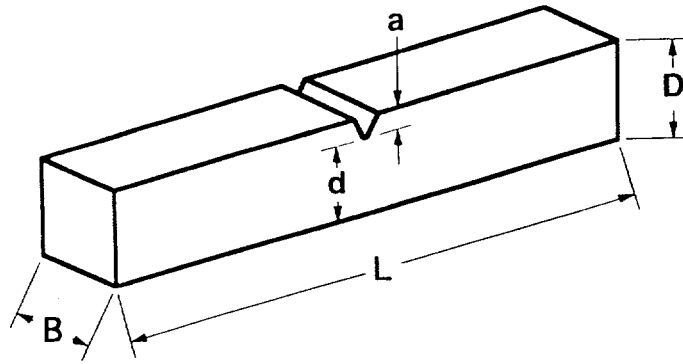
Composition, wt % (standard deviation)									
C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V
0.098 (0.007)	1.56 (0.026)	0.005 (0.0004)	0.005 (0.0006)	0.45 (0.028)	0.25 (0.006)	0.60 (0.006)	0.58 (0.009)	0.31 (0.010)	0.003 (0.0001)

The CVN specimens used in this study were slightly smaller in one cross-section dimension than the standard full-size specimens. This slightly smaller dimension was dictated by the space available in the Fifth Irradiation Series capsules.<sup>7</sup> A typical arrangement of these specimens is shown in Figure 1. The dimension normal to the notch of the undersize CVN specimens is 95% of the full-size specimens, as shown in Figure 2.

The results of CVN impact testing of unirradiated undersize CVN specimens are compared with those of full-size specimens in Figure 3. Each of the curves shown is a hyperbolic tangent fit by nonlinear regression to the experimental results of testing 55 and 85 specimens for the undersize and full-size specimens respectively. Although the slope of the curves in the transition region for the undersize specimens is somewhat steeper than that for the full-size specimens, the transition temperatures at the 41-J level ( $TT_{41-J}$ ) are approximately equal. As is shown later, this difference in slope was not apparent in testing a smaller number of specimens (i.e., 10 to 18) of either the irradiated or irradiated and annealed specimens of nominally the same geometry. Thus, this difference in slope may be due to the sensitivity of the undersize specimen to variations in the notch geometry that are smaller than the tolerances specified by ORNL for all CVN specimens. [The tolerances are considerably tighter than those specified in ASTM Recommended Method for Notched Bar Impact Testing of Metallic Materials (E 23-86). These tighter requirements are dictated by the heat transfer considerations for specimens to be included in irradiation capsules.]



**Figure 1. Undersize Charpy V-notch specimens irradiated in the available space of the Fifth Irradiation Series capsule.**



	L (in) (mm)	B (in) (mm)	D (in) (mm)	d (in) (mm)	a (in) (mm)	a/D
Undersized specimens	2.165 55.00	0.394 10.00	0.375 9.52	0.303 7.71	0.072 1.82	0.191
Standard specimens	2.165 55.00	0.394 10.00	0.394 10.00	0.315 8.00	0.079 2.00	0.200

Figure 2. Dimensions of the undersize Charpy V-notch specimens compared with standard full-size specimens.

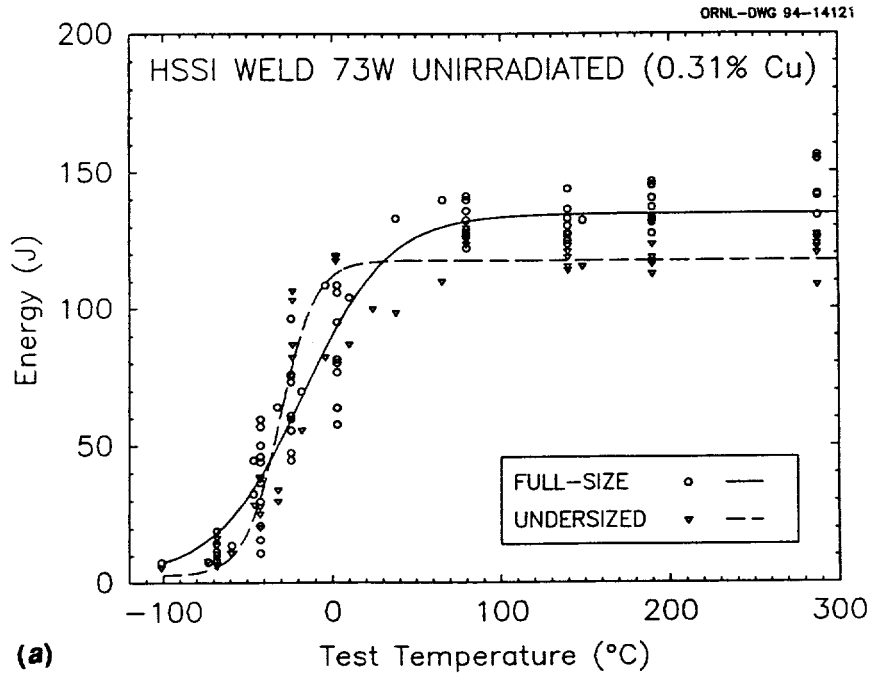
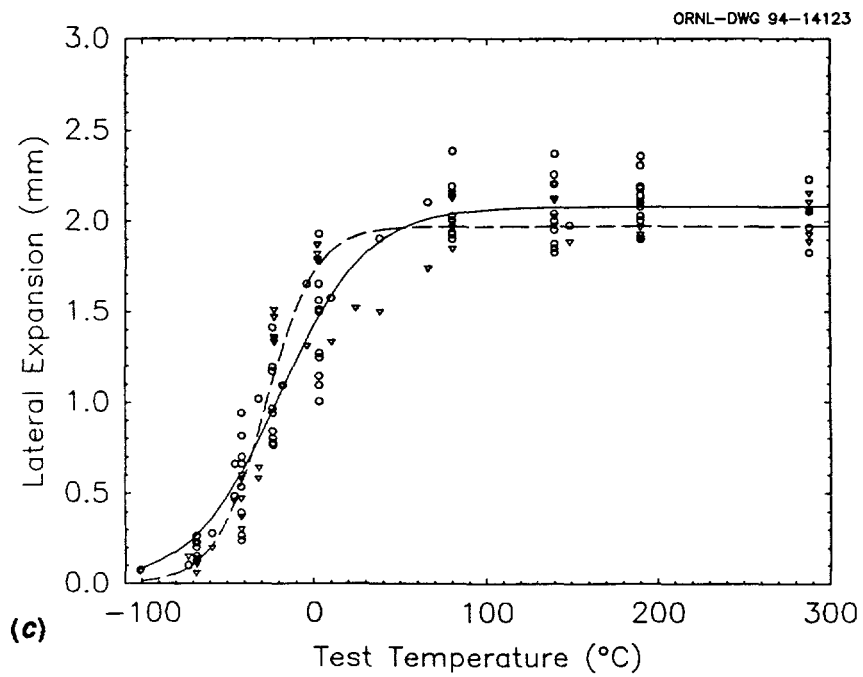
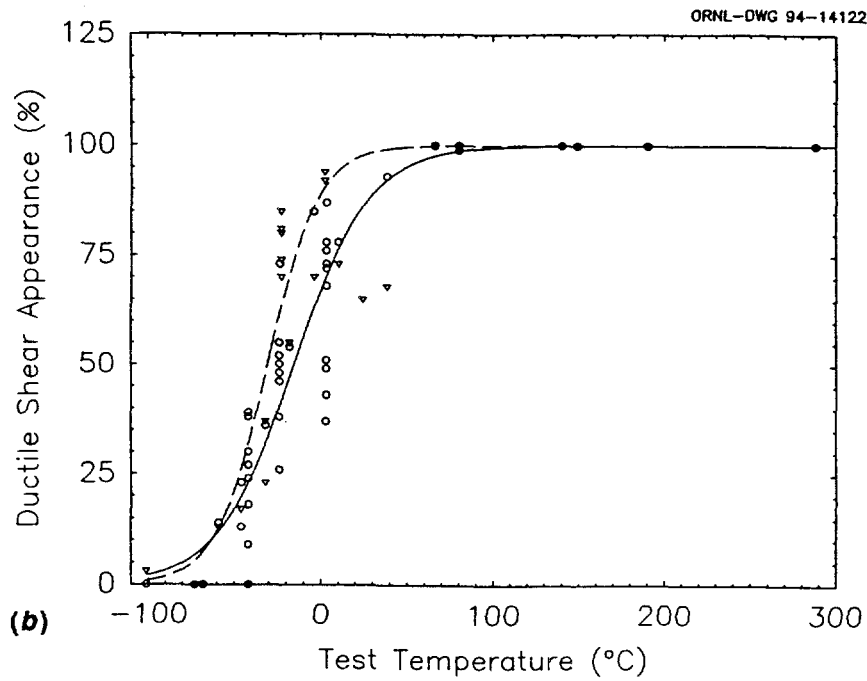


Figure 3. Comparison of the Charpy V-notch impact energy of the unirradiated weld 73W specimens with full-size specimens: (a) energy, (b) ductile appearance, and (c) lateral expansion.



**Figure 3 (Cont.)**

An initial study of the effect of thermal annealing on recovery of fracture toughness of LUS submerged-arc welds was performed on compact specimens irradiated under the Second and Third Irradiation Series. As has been previously described,<sup>8-9</sup> the seven submerged-arc welds, identified as HSSI 61W through 67W, were fabricated with materials and procedures used in early pressurized-water RPVs. These welds have average copper levels ranging from 0.21 to 0.42%; nickel levels were around 0.6% and, thus, were considered "sensitive" to neutron irradiation embrittlement and typical of some early pressure vessel welds. The use of Linde 80 flux produced welds with large contents of very small inclusions, leading to relatively low Charpy upper-shelf energies. The chemical compositions of the welds are presented in Table 4.

The compact specimens of 12.7-cm (0.5T-CT) and 20.3-mm (0.8T-CT) thickness were used for J-R curve characterization of irradiated LUS welds 61W, 63W, 64W, 65W, 66W, and 67W. Unfortunately, the design of the already irradiated specimens did not allow for placement of the clip gage on the load line. To overcome this

**Table 4. Chemical composition of HSSI welds 61W through 67W**

Weld	Average* composition (wt %)									
	C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V
61W	0.09	1.48	0.020	0.014	0.57	0.16	0.63	0.37	0.23	0.005
	<u>0.07</u>	<u>1.45</u>	<u>0.018</u>	<u>0.014</u>	<u>0.55</u>	<u>0.16</u>	<u>0.62</u>	<u>0.36</u>	<u>0.26</u>	<u>0.005</u>
	0.10	1.52	0.021	0.015	0.58	0.17	0.64	0.38	0.31	0.005
62W <sup>b</sup>	0.083	1.51	0.016	0.007	0.59	0.120	0.537	0.377	0.210	0.010
	<u>0.078</u>	<u>1.41</u>	<u>0.013</u>	<u>0.007</u>	<u>0.55</u>	<u>0.067</u>	<u>0.490</u>	<u>0.365</u>	<u>0.160</u>	<u>0.010</u>
	0.088	1.61	0.020	0.008	0.63	0.173	0.585	0.390	0.260	0.011
62WA	0.083	1.51	0.013	0.007	0.59	0.075	0.510	0.377	0.200	0.010
	<u>0.078</u>	<u>1.41</u>	<u>0.012</u>	<u>0.007</u>	<u>0.55</u>	<u>0.070</u>	<u>0.480</u>	<u>0.365</u>	<u>0.140</u>	<u>0.010</u>
	0.088	1.61	0.014	0.008	0.63	0.090	0.540	0.390	0.270	0.011
62WB	0.083	1.51	0.018	0.007	0.59	0.170	0.550	0.377	0.177	0.011
	<u>0.078</u>	<u>1.41</u>	<u>0.016</u>	<u>0.007</u>	<u>0.55</u>	<u>0.160</u>	<u>0.540</u>	<u>0.365</u>	<u>0.160</u>	<u>0.010</u>
	0.088	1.61	0.020	0.008	0.63	0.180	0.600	0.390	0.230	0.012
63W	0.098	1.65	0.016	0.011	0.630	0.095	0.685	0.427	0.299	0.007
	<u>0.088</u>	<u>1.62</u>	<u>0.015</u>	<u>0.010</u>	<u>0.580</u>	<u>0.073</u>	<u>0.663</u>	<u>0.415</u>	<u>0.272</u>	<u>0.006</u>
	0.109	1.67	0.017	0.013	0.675	0.118	0.707	0.440	0.326	0.008
64W	0.085	1.59	0.014	0.015	0.520	0.092	0.660	0.420	0.350	0.006
	<u>0.070</u>	<u>1.54</u>	<u>0.012</u>	<u>0.014</u>	<u>0.445</u>	<u>0.074</u>	<u>0.600</u>	<u>0.410</u>	<u>0.310</u>	<u>0.005</u>
	0.100	1.64	0.017	0.016	0.600	0.110	0.720	0.430	0.390	0.008
65W	0.080	1.45	0.015	0.015	0.480	0.088	0.597	0.385	0.215	0.006
	<u>0.070</u>	<u>1.42</u>	<u>0.014</u>	<u>0.013</u>	<u>0.450</u>	<u>0.076</u>	<u>0.585</u>	<u>0.370</u>	<u>0.180</u>	<u>0.005</u>
	0.090	1.49	0.017	0.017	0.610	0.100	0.610	0.400	0.250	0.008
66W	0.092	1.63	0.018	0.009	0.540	0.105	0.595	0.400	0.420	0.009
	<u>0.075</u>	<u>1.59</u>	<u>0.017</u>	<u>0.009</u>	<u>0.480</u>	<u>0.090</u>	<u>0.580</u>	<u>0.380</u>	<u>0.350</u>	<u>0.007</u>
	0.010	1.67	0.020	0.010	0.600	0.120	0.610	0.420	0.490	0.012
67W	0.082	1.44	0.011	0.012	0.500	0.089	0.590	0.390	0.265	0.007
	<u>0.070</u>	<u>1.40</u>	<u>0.010</u>	<u>0.012</u>	<u>0.410</u>	<u>0.067</u>	<u>0.580</u>	<u>0.370</u>	<u>0.220</u>	<u>0.005</u>
	0.095	1.48	0.013	0.013	0.590	0.110	0.600	0.410	0.310	0.010

\*Top entry is the average value, while numbers shown below each entry indicate the range of composition measurements.

<sup>b</sup>Weld 62W is a duplex weld with designations 62WA and 62WB.

problem, razor blades were attached to the front face of the specimens (i.e., crack mouth) by means of spot welding. A special procedure (i.e., technology) of spot welding in the hot cell was developed. Thus, the measurement of crack-mouth displacement was used to develop a J-R curve. The front face displacements were converted to load-line displacements using a geometric correction factor that accounted for the specimen center of rotation. The specimens of welds 61W and 63W tested were side-groove 5% on each side; all other specimens tested, 10% on each side.

## 6.2 Material Conditions Investigated

The CVN specimens of HSST weld 73W were annealed at 343 and 454°C (650 and 850°F). These two temperatures have often been investigated as lower and upper bounds of possible annealing temperatures.<sup>10</sup> The 343°C (650°F) temperature could be used for a wet anneal, which is considerably simpler to perform, since the reactor internals do not have to be removed, than a dry anneal at 454°C (850°F). A 168-h anneal was investigated for the lower temperature, and when the recovery was insignificant, no other annealing times were investigated. Four annealing times varying from 1 day to 2 weeks (336 h) at 454°C (850°F) were investigated. One of the results of this investigation is that annealing increased the upper-shelf energy to values greater than those of the unirradiated specimens. This was not unexpected since other investigators have also noted such an effect. Annealing and aging effects are generally considered rate processes where the temperature is the dominant parameter and time is of secondary influence. As part of the original fabrication procedure, the 73W weld was postweld heat-treated at 607°C (1125°F) for 40 h; thus, exposure at the lower temperature for 168 h would not be expected to have significant effect. To determine whether this increase in upper-shelf energy was associated with the irradiated condition, the unirradiated specimens are also being aged at 460 and 490°C (860 and 914°F). The conditions investigated have been summarized in Table 5.

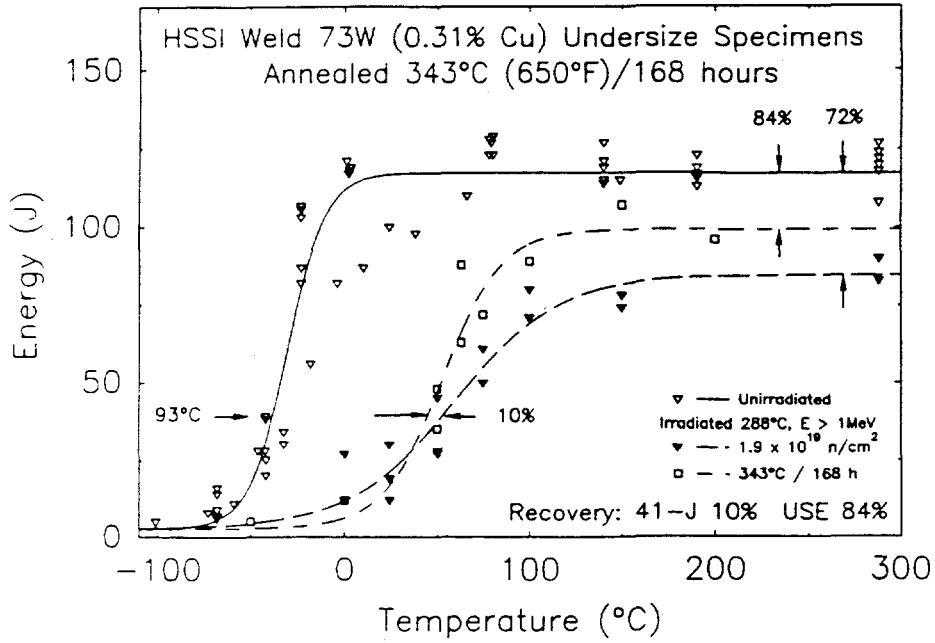
**Table 5. Material conditions investigated**

Material Condition	Aging or annealing time (h)
Unirradiated	
Unirradiated and aged at 460°C (860°F)	168
Unirradiated and aged at 490°C (914°F)	168
Irradiated	
Annealed at 343°C (694°F)	168
Annealed at 454°C (850°F)	24, 96, 168, 336

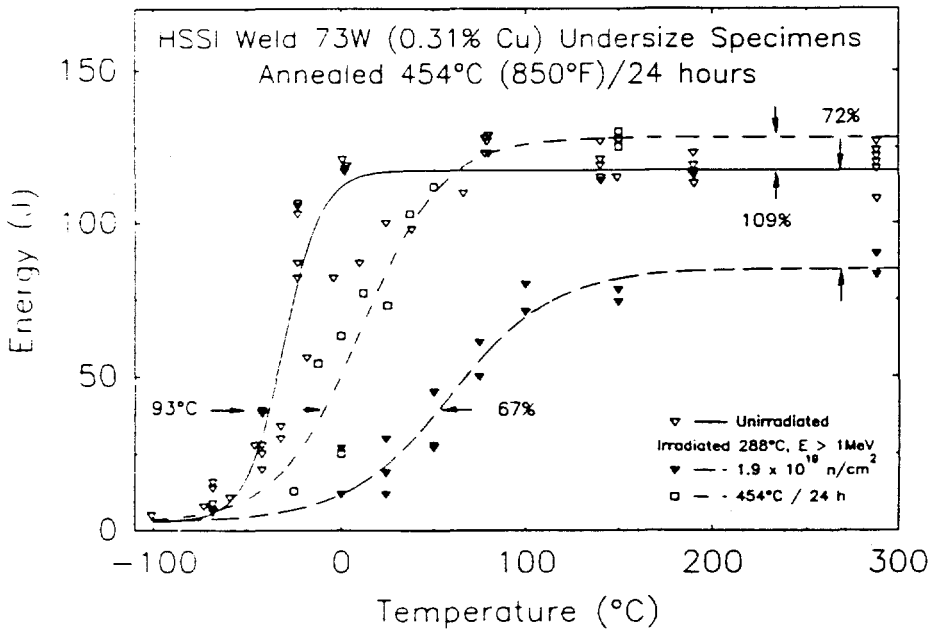
The compact specimens of LUS welds 61W through 67W had been previously irradiated at the ORNL Bulk Shielding Reactor. Each weld was irradiated to a certain value of neutron fluence in the range from 0.5 to  $1.3 \times 10^{19}$  neutrons/cm<sup>2</sup> (>1 MeV) in the average temperature range of 275 to 300°C (527 to 572°F). The irradiated specimens were annealed at 454°C (850°F) for 168 h. Fracture toughness tests were performed at temperatures selected to match those of the unirradiated and irradiated tests.

## 6.3 Test Results

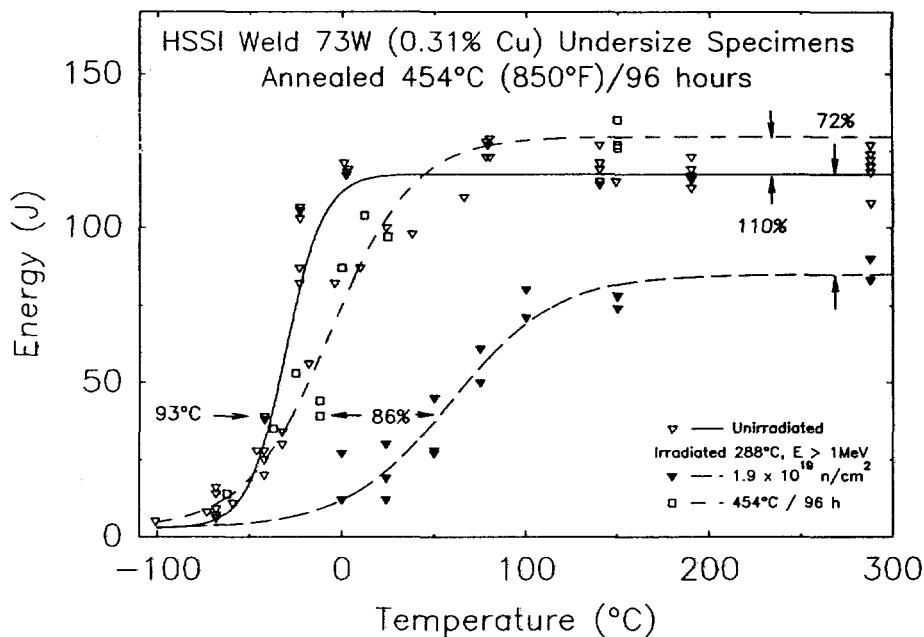
The results of CVN tests on 73W welds for the conditions investigated are shown in Figures 4 through 8. The recovery of CVN impact properties is measured by the changes in values of the upper-shelf energy and TT<sub>41-J</sub> due to annealing when compared with the unirradiated values. The values of the upper-shelf energy and TT<sub>41-J</sub>



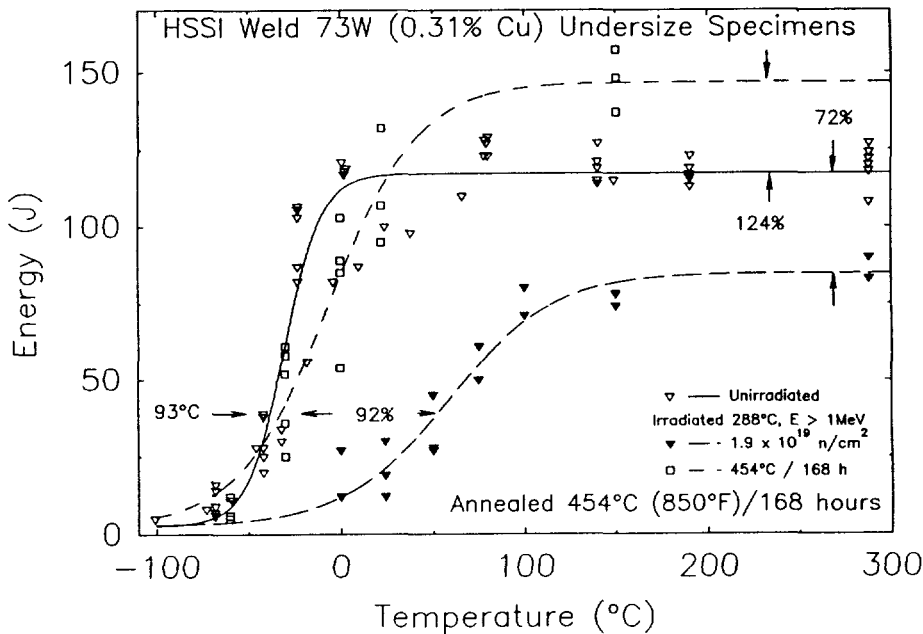
**Figure 4.** Results of testing undersize Charpy V-notch specimens annealed at 343°C (649°F) for 24 h compared with those in the unirradiated and irradiated conditions. The percent recovery is also indicated.



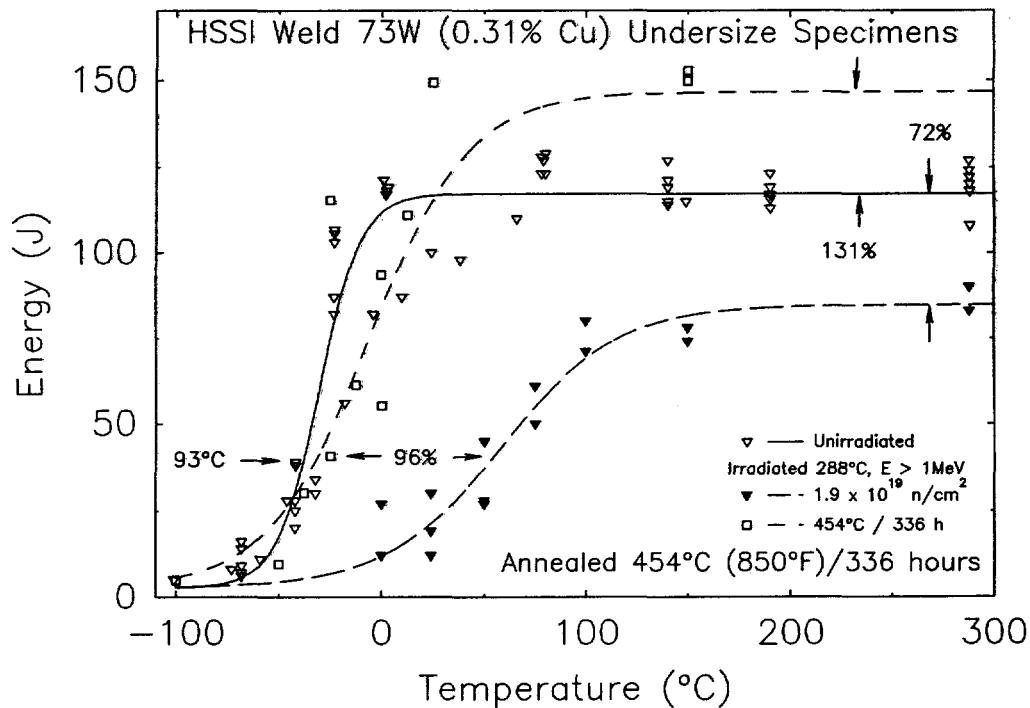
**Figure 5.** Results of testing undersize Charpy V-notch specimens annealed at 454°C (850°F) for 24 h compared with those in the unirradiated and irradiated conditions. The percent recovery is also indicated.



**Figure 6.** Results of testing undersize Charpy V-notch specimens annealed at 454°C (850°F) for 96 h compared with those in the unirradiated and irradiated conditions. The percent recovery is also indicated.



**Figure 7.** Results of testing undersize Charpy V-notch specimens annealed at 454°C (850°F) for 168 h compared with those in the unirradiated and irradiated conditions. The percent recovery is also indicated.



**Figure 8. Results of testing undersize Charpy V-notch specimens annealed at 454°C (850°F) for 336 h compared with those in the unirradiated and irradiated conditions. The percent recovery is also indicated.**

were calculated from a nonlinear regression fit of a hyperbolic tangent equation to CVN impact energy. The hyperbolic tangent equation was also fitted to the experimental values of percent ductile shear appearance and lateral expansion and is of the form:

$$Y = \frac{US + LS}{2} + \frac{US - LS}{2} \operatorname{Tanh} \left( \frac{T - MTT}{TZW/2} \right) \quad (1)$$

where

- y = energy, percent ductile shear appearance, or lateral expansion,
- US, LS = upper- and lower-shelf values, respectively.
- T = test temperature,
- MTT = mid-transition temperature,
- TZW = transition zone width.

In case of fitting energy values, the LS was prescribed to be 2.7 J, the average value obtained by testing five submerged-arc welds at liquid nitrogen temperature of -196°C (321°F).<sup>6</sup> For the lateral expansion and percent ductile shear appearance, the value of LS was prescribed to be zero. The upper-shelf value, US, of ductile shear was prescribed to be 100%.

The percent recovery of the  $TT_{41-J}$  is defined as the ratio of the residual transition temperature shift after annealing to the shift due to irradiation,  $\Delta TT_{41-J}$ , or:

$$\% \text{ Recovery } TT_{41-J} = \frac{[TT_{41-J}]_{irr} - [TT_{41-J}]_{ann}}{[TT_{41-J}]_{irr} - [TT_{41-J}]_{unir}} \times 100, \quad (2)$$

where  $TT_{41-J}$  is the transition temperature at the 41-J level for the condition indicated by the outer subscript.

The percent recovery of the upper-shelf energy is defined as the ratio of the values of the USE in the irradiated and annealed condition to the unirradiated condition:

$$\% \text{ USE Recovery} = \frac{[USE]_{ann}}{[USE]_{unir}} \times 100 \quad (3)$$

Except for the 343°C (650°F) annealing temperature, which did not show any significant recovery as mentioned previously, the degree of CVN impact energy recovery was dependent on the length of annealing time. The  $TT_{41-J}$ , upper-shelf energy, change, and percentage recovery for each of the conditions investigated has been summarized in Table 6. The percentage recovery of both the  $TT_{41-J}$  and the upper-shelf energy has been plotted in Figure 9 for annealing at 454°C (850°F).

The values for J-integral were determined using both the procedure specified in ASTM Standard Test Method for Determining J-R Curves (E 1152) and a modified version of the J-integral as proposed by Ernst.<sup>11</sup> Modified J is used here because this version of the J-integral was chosen and reported in the previous study for the unirradiated and irradiated conditions.<sup>8</sup> The values of tearing modulus were determined as:

$$T_{avg} = \frac{E}{\sigma_f^2} \cdot \frac{dJ}{da}, \quad (4)$$

where E is the modulus of elasticity,  $\sigma_f$  is the flow stress (the average of the yield and ultimate strength), and  $dJ/da$  is the average slope of the J-R curve between the exclusion lines.

Figures 10 and 11 present J-R curves from 0.5 TC(T) specimens of welds 66W and 67W, respectively, after annealing at 454°C (850°F) for 168 h. For comparison, J-R curves of 0.5 TC(T) specimens of these welds in the unirradiated and irradiated conditions obtained in the previous study<sup>8</sup> are also plotted. Table 7 lists the values of  $J_{IC}$  and  $T_{avg}$  obtained for all six welds in the annealed condition. Figures 12 through 17 summarize all the data for the studied welds in the unirradiated, irradiated, and annealed conditions.

## 6.4 Discussion and Conclusions

Annealing the weld 73W at 454°C (850°F) for various length of time from 24 to 336 h has recovered the upper-shelf energy to values that exceeded the unirradiated welds, which has also been observed in other investigations. As mentioned previously, this increase cannot be explained by the diffusion process rate theory in which temperature is the dominant parameter, since the weld was already postweld heat-treated at the higher temperature of 607°C (1125°F). Assuming the same mechanisms are operative, the annealing at 454°C (850°F) should not produce any changes in impact energy. Thus, the results imply a different mechanism at the lower temperature. Aging of unirradiated weld 73W at 460 and 490°C (860 and 914°F) for 168 h also increased the

**Table 6. Values of the 41-J transition temperature and upper-shelf energy for all conditions investigated (blank values are unavailable or not applicable)**

Condition	Annealing/aging		41-J transition temperature			Upper-shelf energy		
	Temperature (°C)	Time (h)	Value (°C)	Change <sup>a</sup> (J)	Recovery <sup>b</sup> (J)	Value (J)	Change <sup>c</sup> (J)	Changed <sup>d</sup> (%)
Unirradiated			-38			118		
Irradiated			55	93		85	-33	72
Irradiated/annealed	343	168	46	84	10	99	-19	84
Irradiated/annealed	454	24	-7	31	67	128	10	108
Irradiated/annealed	454	96	-25	13	86	130	12	110
Irradiated/annealed	454	168	-31	7	92	147	29	125
Irradiated/annealed	454	336	-34	4	96	154	36	131
Unirradiated/aged	460	168				138		
Unirradiated/aged	460	168				138		

<sup>a</sup>Change from unirradiated value; positive values indicated an increase in the transition temperature.

<sup>b</sup>Defined by Eq. (2).

<sup>c</sup>Change from unirradiated value; positive values indicate an increase in upper shelf.

<sup>d</sup>Defined by Eq. (3).

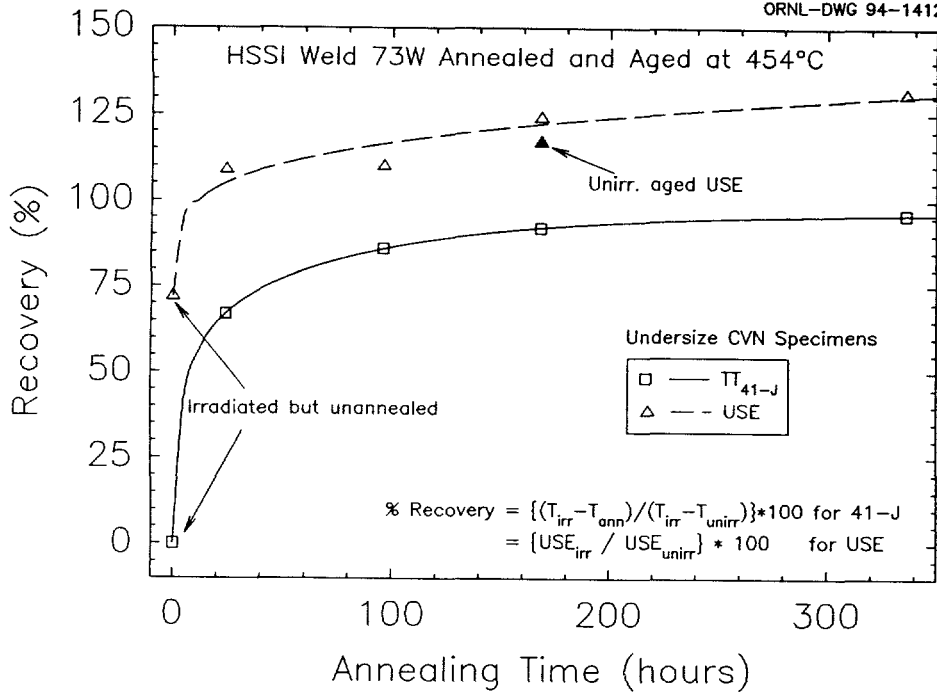


Figure 9. Summary of the percent recovery of the 41-J level transition temperature and the upper-shelf energy following thermal annealing.

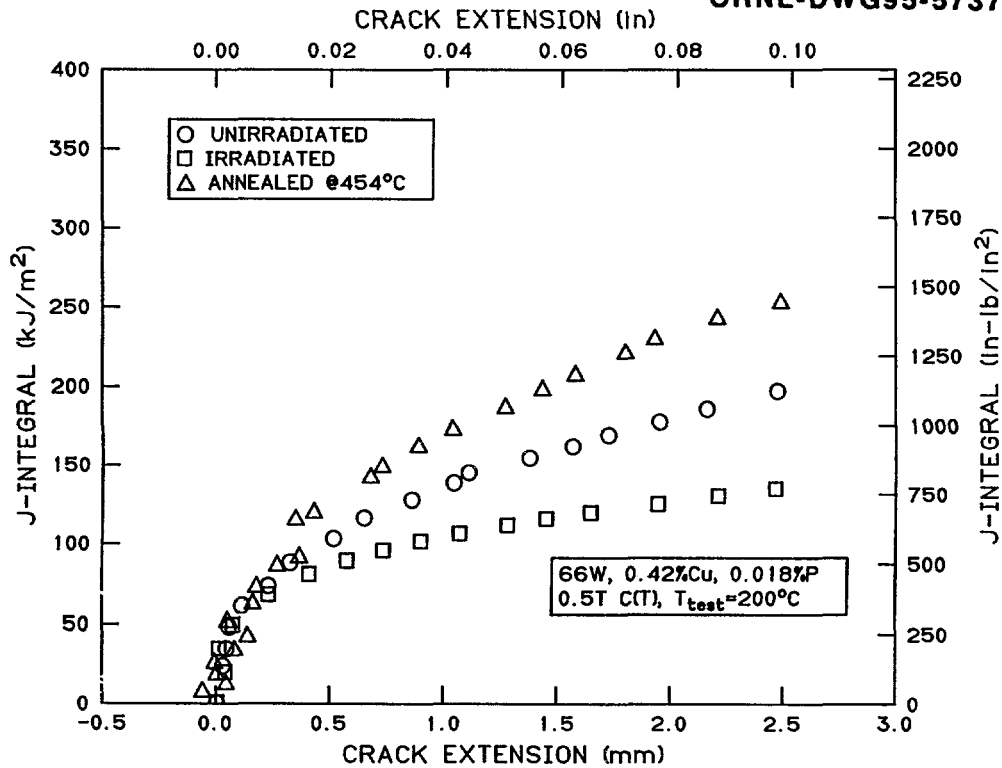


Figure 10. J-R curves of 0.5 TC (T) specimens of weld 66W tested at 200°C (392°F) in unirradiated, irradiated, and annealed conditions.

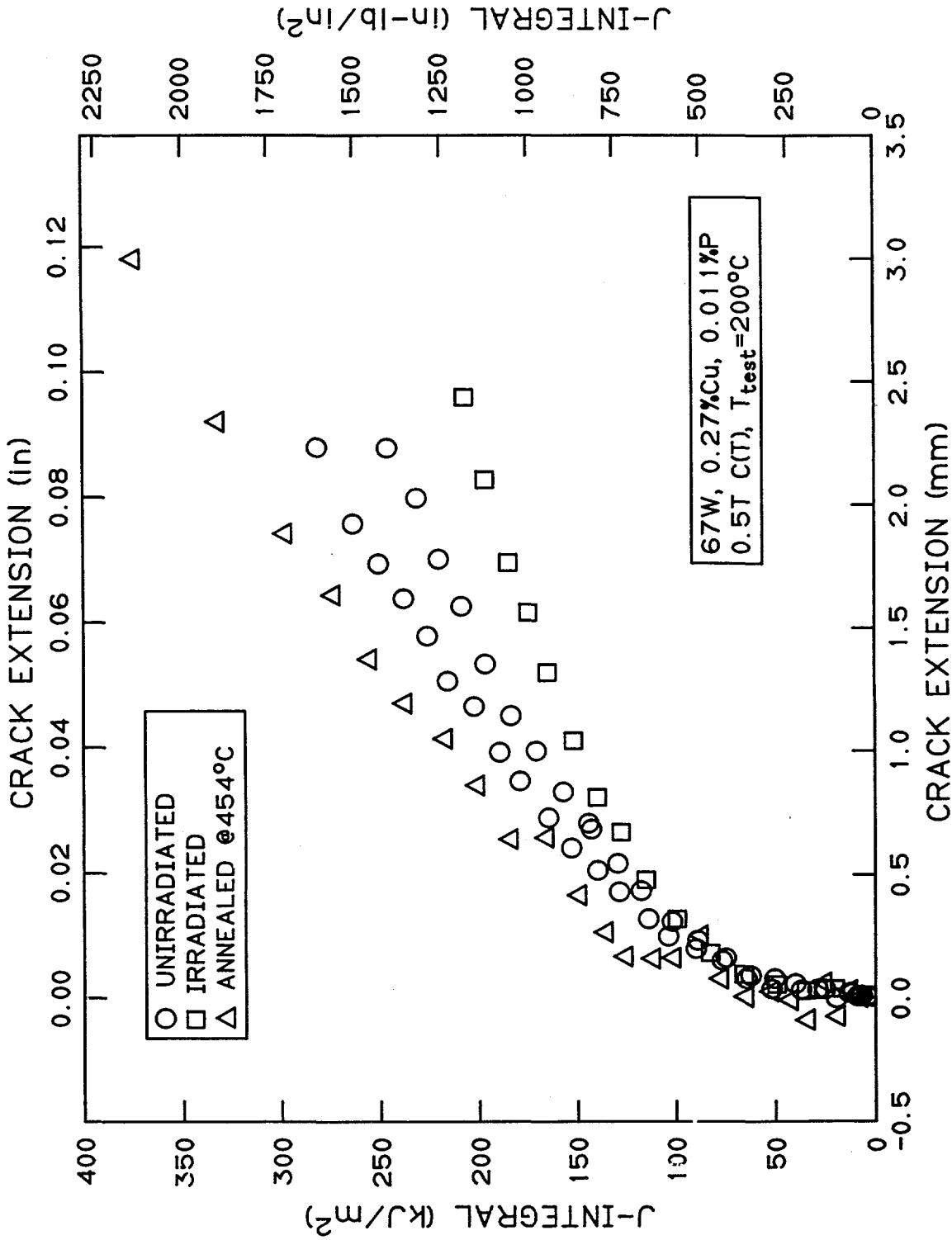


Figure 11. J-R curves of 0.5 TC (T) specimens of weld 67W tested at 200°C(392°F) in unirradiated, irradiated, and annealed conditions.

Table 7. Values of  $J_{IC}$  and  $T_{avg}$  of studied welds after annealing at 454°C (850°F) for 168 h

Weld	Specimen size (T)	$T_{irr}$ (°C)	Fluence ( $10^{18}$ n/cm <sup>2</sup> )	$T_{test}$ (°C)	$J_{IC}$ (kJ/m <sup>2</sup> )		$T_{avg}$	
					E 1152	Modified	E 1152	Modified
61W	0.8	303	12.0	121	150	152	51	57
63W	0.5	291	8.3	171	167	171	131	155
64W	0.8	284	7.7	177	226	234	139	156
	0.5	286	5.7	177	111	114	75	87
65W	0.5	286	6.5	200	118	122	74	88
66W	0.5	279	8.0	200	90	92	37	42
67W	0.5	278	3.8	200	126	129	72	89

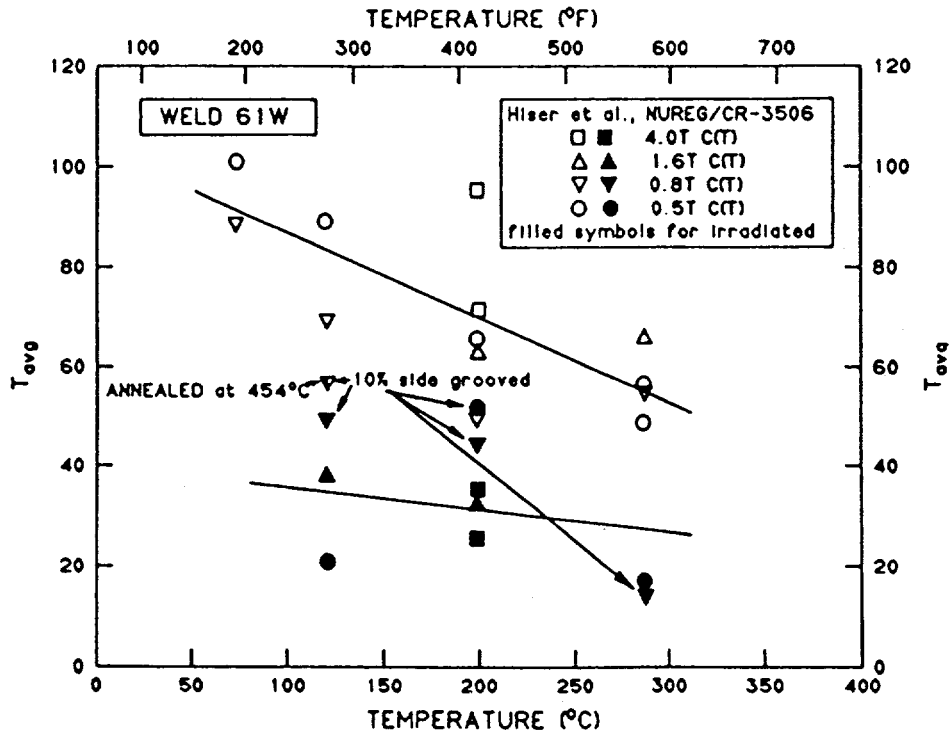
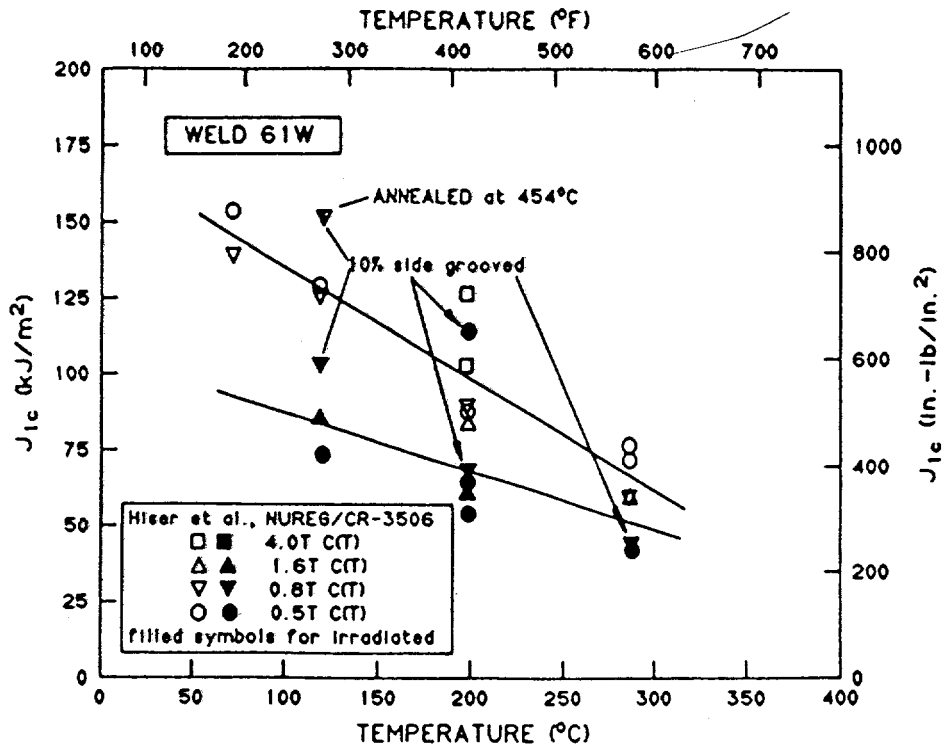


Figure 12. Values of  $J_{1c}$  (top) and  $T_{avg}$  (bottom) of 61W in unirradiated, irradiated, and annealed conditions.

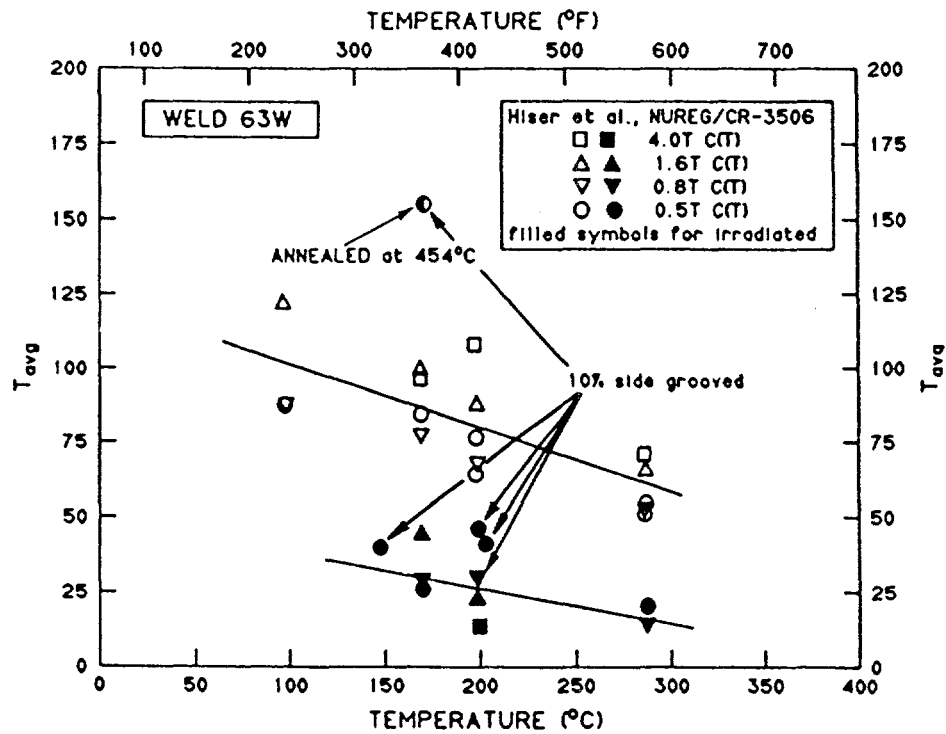
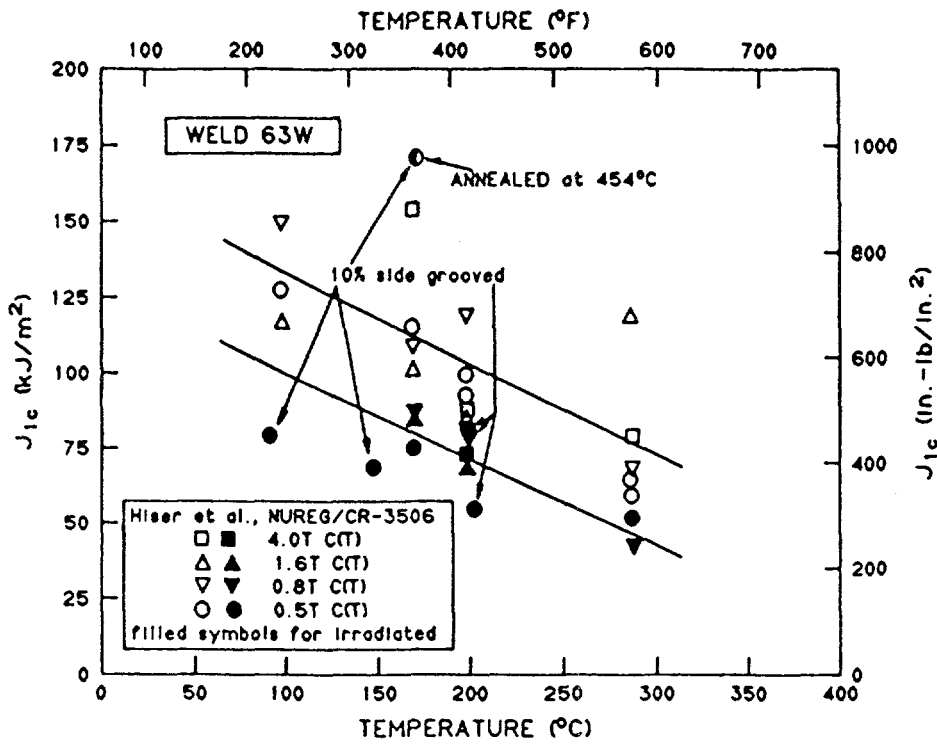


Figure 13. Values of  $J_{1c}$  (top) and  $T_{avg}$  (bottom) of 63W in unirradiated, irradiated, and annealed conditions.

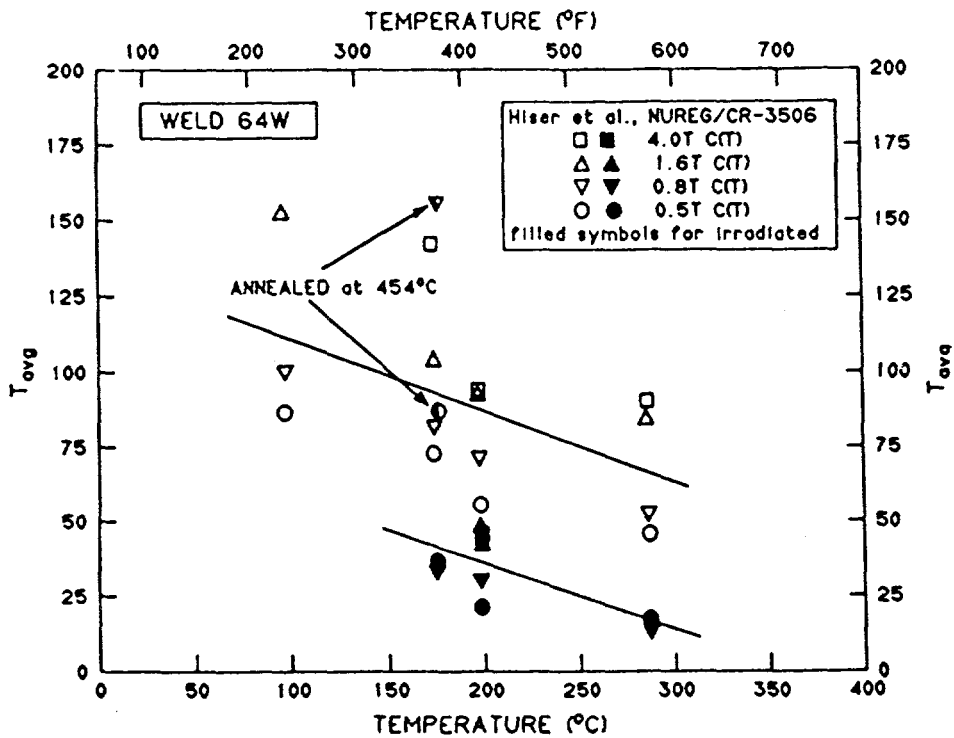
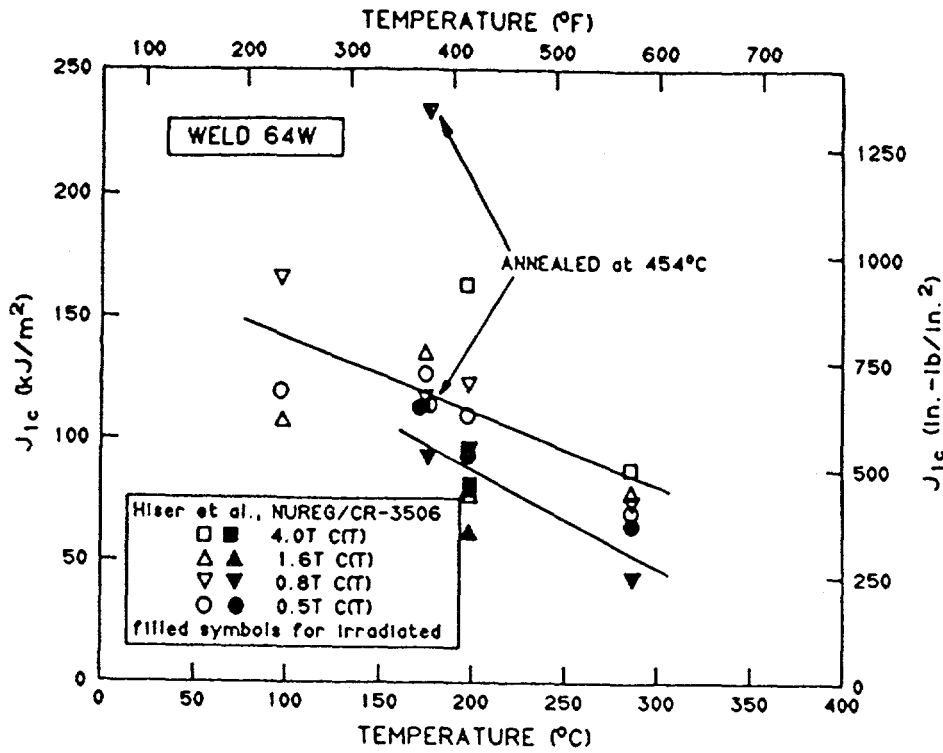


Figure 14. Values of  $J_{1c}$  (top) and  $T_{avg}$  (bottom) of 64W in unirradiated, irradiated, and annealed conditions.

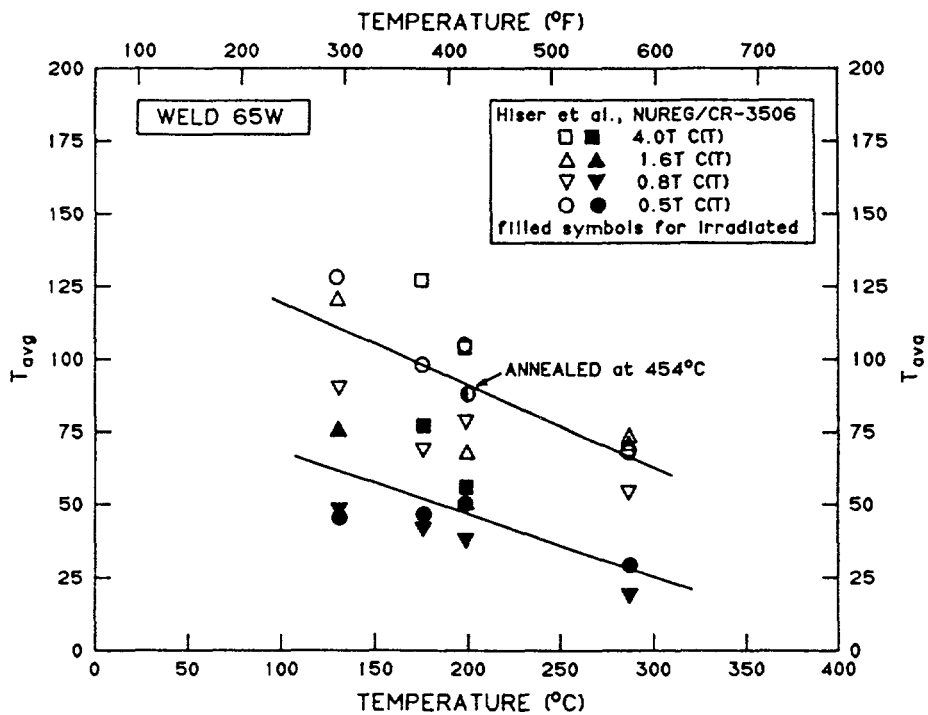
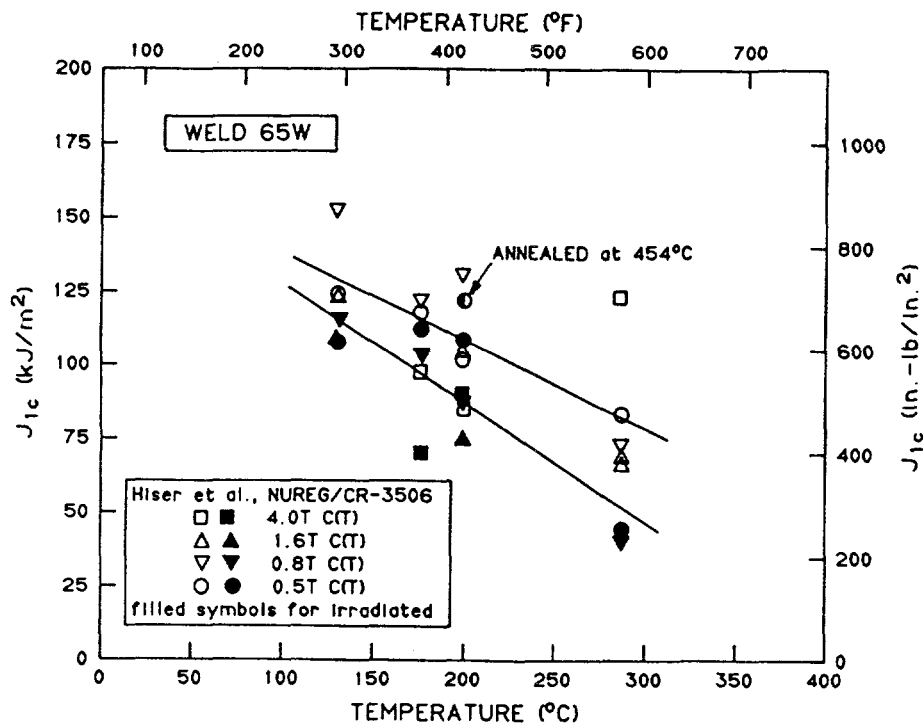


Figure 15. Values of  $J_{1c}$  (top) and  $T_{avg}$  (bottom) of 65W in unirradiated, irradiated, and annealed conditions.

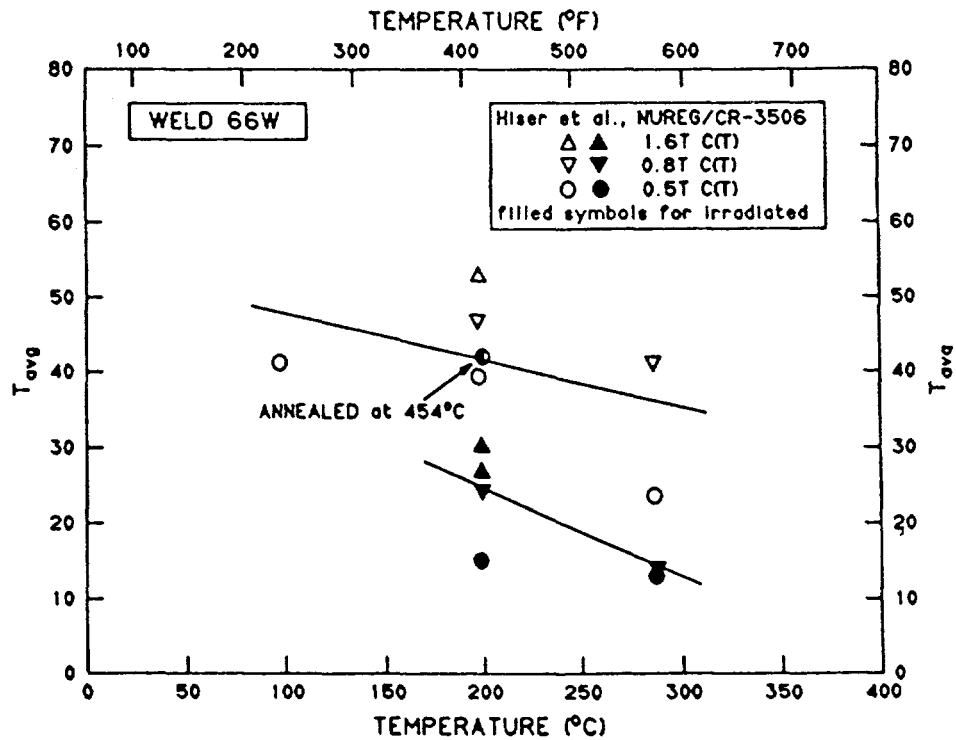
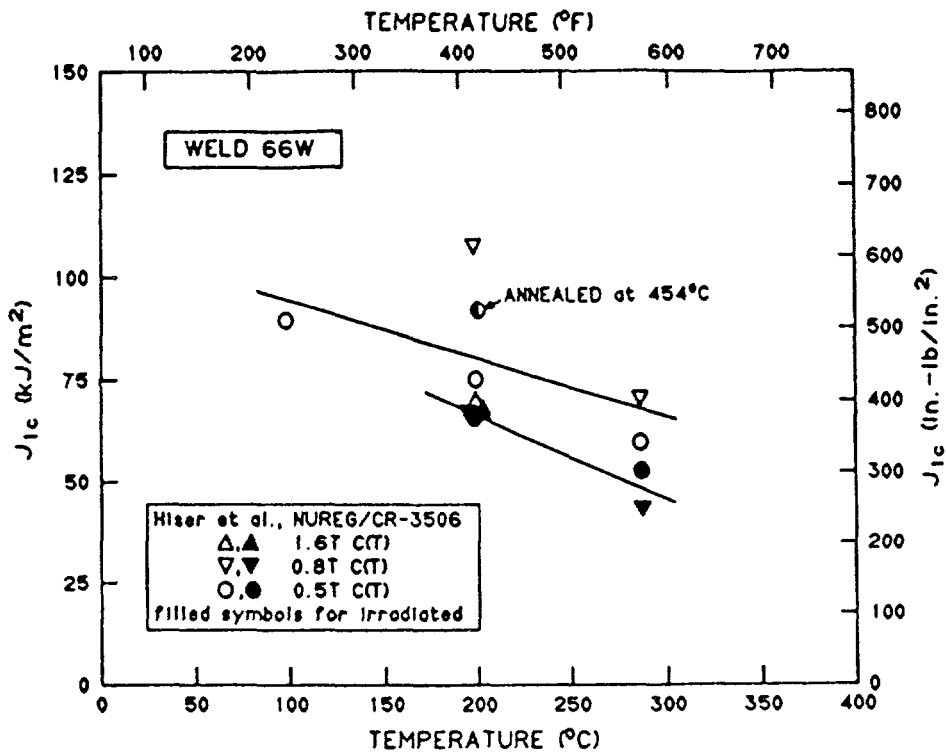


Figure 16. Values of J<sub>1c</sub> (top) and T<sub>avg</sub> (bottom) of 66W in unirradiated, irradiated, and annealed conditions.

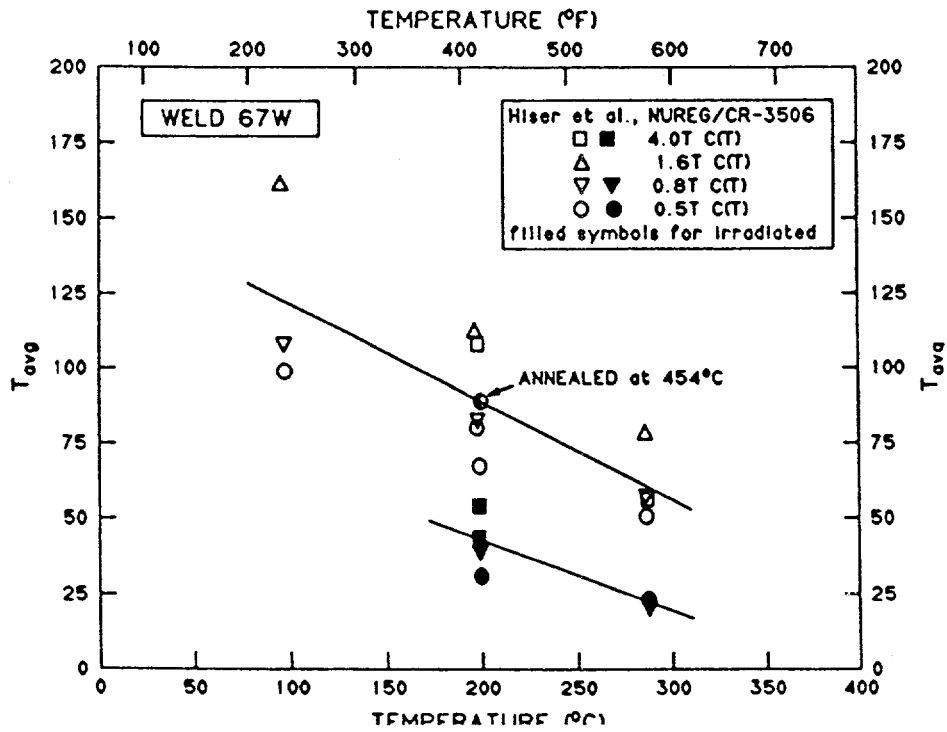
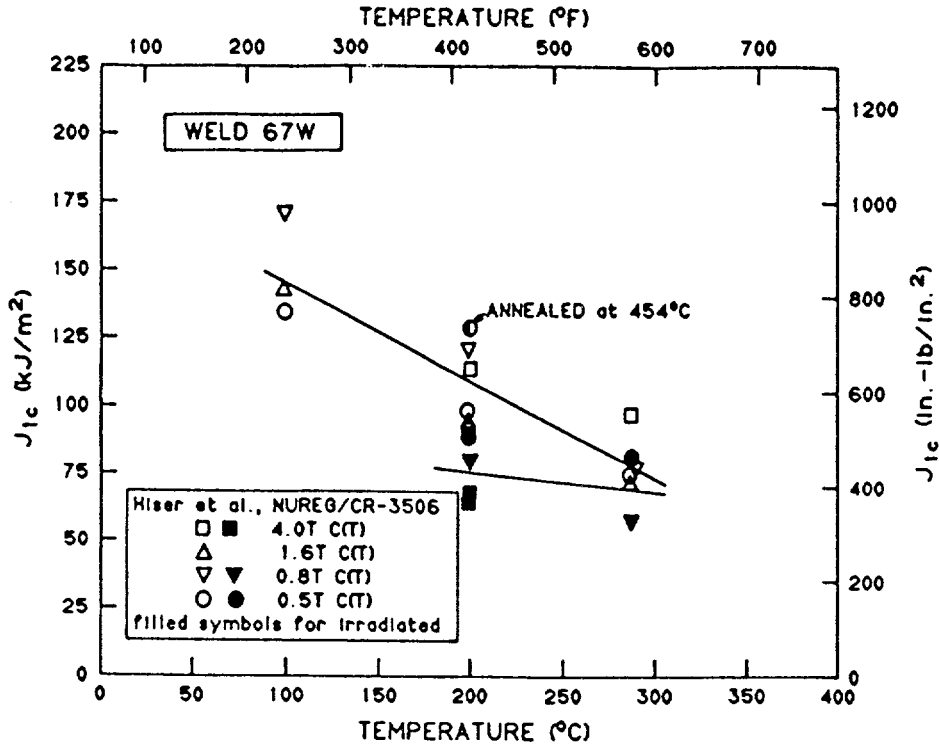
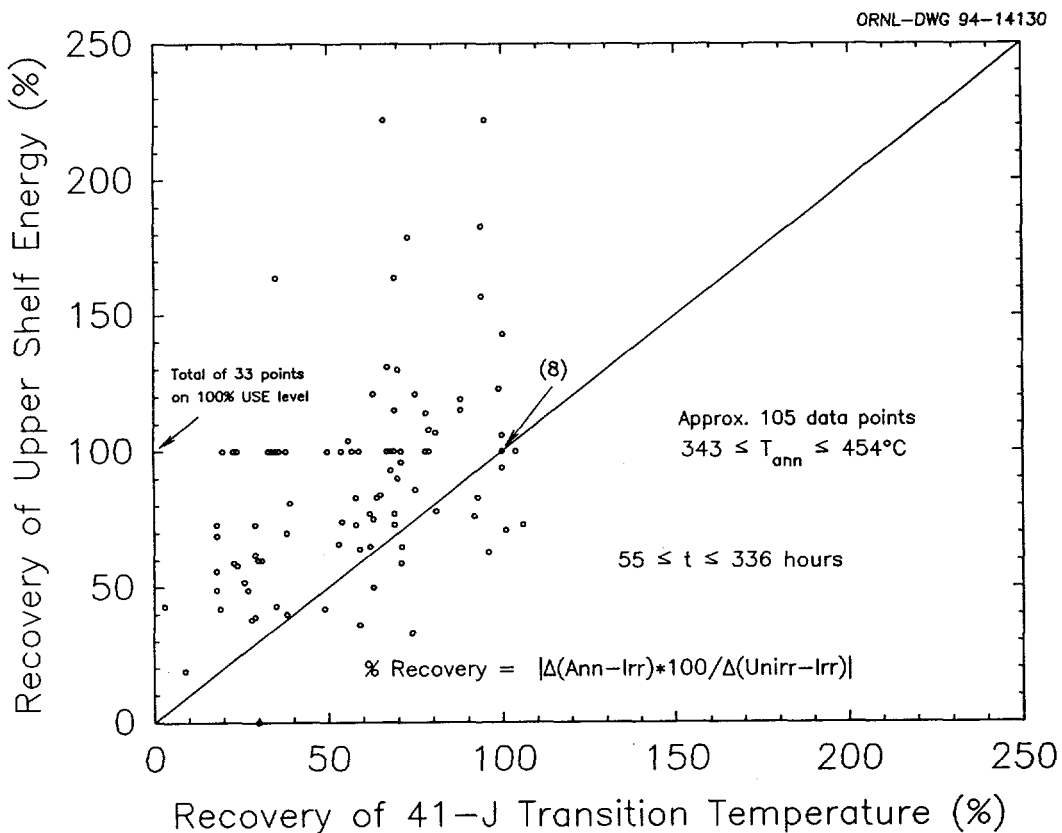


Figure 17. Values of  $J_{1c}$  (top) and  $T_{avg}$  (bottom) of 67W in unirradiated, irradiated, and annealed conditions.

value of upper-shelf energy; therefore, this increase could not be explained solely by changes in the material due to irradiation. Note that the aging of unirradiated weld 73W did not cause any noticeable changes in the value of TT<sub>41-J</sub>.

Annealing weld 73W at 454°C (850°F) for 24 h recovered about two-thirds of the transition temperature shift caused by neutron irradiation. Annealing at this temperature for longer times increases the recovery but at a decreasing rate; doubling the annealing time from 168 to 336 h in increases the percent recovery from 92 to 96%, which may not be a reasonable return for the extra annealing time invested.

In all the above cases of annealing of irradiated weld 73W at 454°C (850°F), the percentage recovery of the upper-shelf energy was greater than the percentage recovery of TT<sub>41-J</sub>. This was also observed in other investigations, as can be shown by plotting the percent recovery of upper shelf vs the percent recovery of the TT<sub>41-J</sub> for RPV steels. The data in Figure 18, which have been extracted from the ORNL Test Reactor Embrittlement Data Base,<sup>12</sup> show that the majority of the data lie above the straight line with a 1:1 slope. This confirms the results from this investigation (i.e.) the upper-shelf energy recovers faster than the TT<sub>41-J</sub>, and the percentage recovery of upper-shelf energy as defined by Eq. (3) is greater than 100%. This result can be useful in cases for which the number of specimens available is less than desired for an adequate determination of both TT<sub>41-J</sub> and upper-shelf energy. More specimens could be devoted to determine the TT<sub>41-J</sub>, and a smaller number than desirable could be used to confirm the recovery of the upper-shelf energy.



**Figure 18. Percent recovery of the upper-shelf energy vs percent recovery of the 41-J level transition temperature shift following thermal annealing. The solid line has a 1:1 slope. The other lines are the 95% confidence limits on the mean and predicted. Data are extracted from ref. 12.**

The data show that in all cases annealing at 454°C (850°F) produced a very positive effect on the J-R curve recovery of irradiated high-copper, LUS welds. A similar result was observed for recovery of CVN properties. Annealing at 454°C (850°F) resulted in recovery of the upper-shelf energy and  $J_{IC}$  to well above unirradiated values, while the transition temperature,  $TT_{41-J}$ , and tearing modulus,  $T_{avg}$ , recovered to the unirradiated levels only.

The rate of toughness degradation of irradiated and annealed RPV steels is the present focus of investigations at ORNL. This is a major consideration in determining how long the RPV can be operated safely after annealing. It is possible that no archival material will be available for some of the older RPVs for which annealing will be considered and that the surveillance data from CVN specimens will be the only data available that describes the rate of embrittlement. Thus, relating the rate of toughness degradation due to reirradiation to the rate of transition temperature shift of CVN specimens (the trend curve) from the unirradiated state is important.

## 6.5 Tests on Regulated-Atmosphere Annealing Furnace

Preliminary tests at 150°C (302°F) of the recently purchased temperature controller for the regulated-atmosphere annealing furnace have been completed (this furnace may be used for future annealing work in the hot cell). It was assembled using components that have been used for some time at ORNL and have given good service. It consists of a Research, Inc., Microstar Model 828D controller, a Honeywell UDC2000 high-temperature limit control, and a solid state Control Concepts zero-fired relay. The range in temperature variation of specimens in the furnace must still be verified. The furnace, which is capable of temperatures up to 1200°C (2192°F), requires water cooling of the door flange to protect the elastomeric seal from temperatures greater than 260°C (500°F). The use of cooling water in the hot cell is expensive, and tests will be conducted to see if annealing at 454°C (850°F) can be performed without cooling water.

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## 7. Irradiation Effects in a Commercial Low Upper-shelf Weld (Series 10)

D. E. McCabe, S. K. Iskander, I. I. Siman-Tov, and D. W. Heatherly

### 7.1 Crack-Arrest Toughness Results

The testing of unirradiated Midland beltline WF-70 weld metal for crack-arrest fracture toughness,  $K_{Ia}$ , was marginally successful from the standpoint that the crack arrested in many cases with insufficient remaining ligaments for valid  $K_{Ia}$  by ASTM test method E 1221. The development of valid  $K_{Ia}$  data was considered sufficiently important to retry these tests with a different specimen design. The second specimen design is a duplex crack-arrest specimen where relatively brittle AISI 4340 is used as the crack initiator material. Five such specimens have been fabricated and are now ready for testing. Results will be reported in the next progress report.

### 7.2 Unirradiated Fracture Toughness Results

The fracture toughness tests of unirradiated material have been completed, analyzed, and presented in a draft NUREG report. The report compiles and evaluates the unirradiated material properties of Midland weld WF-70. This has been completed, submitted for peer review, and revised according to comments received. It is currently in the Metals and Ceramics Division Records Office for final preparation prior to publication. The preliminary evaluation of the Midland weld metal using the conventional CVN and drop weight nil-ductility transition temperature tests indicated that the nozzle course WF-70 weld and beltline WF-70 weld metal had essentially the same fracture toughness. In particular, both had a dropweight nil-ductility transition temperature of about  $-55^{\circ}\text{C}$  ( $-67^{\circ}\text{F}$ ), both showed a mid-transition CVN temperature of about  $0^{\circ}\text{C}$  ( $32^{\circ}\text{F}$ ), and both had the same upper-shelf energy of 88 J (65 ft-lb). However, there was a significant difference in copper content (i.e., 0.26 wt % in the beltline weld metal and 0.40 wt % in the nozzle course weld metal). Transition temperatures measured in terms of  $K_{Jc}$  values indicated a significant difference between the two weld metals. The reference temperatures (where the median  $K_{Jc}$  is  $100 \text{ MP}\sqrt{\text{m}}$  with 1T compact specimens) for the beltline and nozzle course welds were  $-60$  and  $-33^{\circ}\text{C}$  ( $-76$  and  $-27^{\circ}\text{F}$ ) respectively. Additionally, a comparison of J-R curves showed lower ductile tearing resistance (J-R curve) in the nozzle course than in the beltline weld. In general, both materials displayed the typical upper-shelf behavior of LUS weld metal.

### 7.3 Material Irradiation and Transportation Activities

Other activities in this task relate to the transport and disassembly of irradiated capsules. Scoping capsule 10.01 consisted of 1/2 T compact, tensile, and CVN specimens of beltline weld metal. Capsule 10.02 contained a similar complement of nozzle course weld specimens. Both capsules were shipped from the University of Buffalo Reactor site to ORNL. These specimens were irradiated to  $5 \times 10^{18}$  neutrons/cm<sup>2</sup>. The specimens have been positioned in the ORNL hot cell for testing. Capsule 10.05 is large, containing 37 1T compact specimens, 36 1/2 T specimens, and about 400 CVN specimens of assorted materials. This capsule has been retained at the Ford Nuclear Reactor site pending the development plans for transport plus resolution of cask transport regulation requirements. After months of searching for an appropriate ORNL hot cell site to make a cask transfer, we were able to identify a suitable location: an open bay area at the ORNL High Flux Isotope Reactor (HFIR) facility. The equipment needed to execute a safe capsule transfer is being fabricated. All the needed preparatory work to secure approval for transport from HFIR to ORNL is under way. In the meantime, large capsule 10.06 is being irradiated at the Ford Nuclear Reactor. The removable dosimetry tubes indicated that the exposure was 50% complete on January 24, 1994, and the capsule has been rotated in the reactor pool. Control conditions have been very good. Temperature variance over time is within  $2.5^{\circ}\text{C}$  ( $36^{\circ}\text{F}$ ) of the control value. Exposure to  $1 \times 10^{19}$  neutrons/cm<sup>2</sup> is expected to be complete by the end of FY 1994.

## 8. Microstructural Analysis of Radiation Effects

R. E. Stoller, P. M. Rice, K. Farrell, and M. K. Miller

### 8.1 Microstructural Modeling

The necessary calculations were completed to permit an evaluation of the relative importance of copper-rich precipitates (CRP) and point defect clusters in RPV embrittlement. The results are described in detail in a NUREG report that was prepared during this reporting period. The primary conclusions of the analysis were:

- 1 point defect clusters can provide a significant degree of matrix hardening under many irradiation conditions;
- 2 copper-rich precipitates are more important at higher temperatures, lower displacement rates, and high doses;
- 3 since both defect types can be important, careful data extrapolation in either flux or fluence requires that the possibility of crossing from copper-rich precipitates to point defect clusters dominated conditions be considered; and
- 4 the influence of neutron flux or displacement rate on the predicted embrittlement is weak below a fast ( $E > 1.0$  MeV) of about  $5 \times 10^{12}$  n/cm<sup>2</sup>/s, corresponding to a displacement rate of about  $5 \times 10^{-9}$  dpa/s.

Point 4 is particularly significant and is illustrated by Figure 19. In this figure, the predicted change in yield strength at a dose of 0.02 dpa is shown as a function of displacement rate for three levels of copper content. If these predictions are confirmed by experiments, it should reduce concerns about the effects of the fluence lead factors currently employed in reactor surveillance programs and permit the use of a broader range of test reactor data.

### 8.2 Experimental Investigations

The results of the ORNL investigation of the embrittlement of the HFIR pressure vessel indicate that an unusually large ratio of the high-energy gamma-ray flux to fast neutron flux is most likely responsible for the apparently accelerated embrittlement. Gamma rays can indirectly cause atomic displacements by first generating high energy electrons by either pair production or Compton scattering. When these electrons have energies above a few hundred kiloelectron volts, they can displace lattice atoms. Such displacements are usually insignificant, since the fluxes of high-energy neutrons and gamma rays are comparable and since the displacement cross section for these electron events is about 1/1000 of that for fast neutrons.

However, a combination of the beryllium reflector and the long water path between the HFIR core and the pressure vessel leads to a fast gamma flux about 10,000 times greater than the fast neutron flux. In this case, more displacements may be generated by the gamma-induced electrons than by the fast neutrons. Figure 20 demonstrates that when all of the atomic displacements are counted, the Charpy shift data from the HFIR surveillance program are well correlated with the data from other sources.

A series of 4-MeV iron ion irradiations was completed on six of the model alloys selected for the initial phase of the microstructural/mechanical correlation work. The alloys, designated VM348, VM350, VM387, VM390, VM397, and VM399, were selected to examine variations in copper, nitrogen, and carbon. Specimens were irradiated at 300°C (572°F) to three peak doses, 0.02, 0.2, and 2.0 dpa, corresponding to fast neutron fluences of about  $8 \times 10^{18}$ ,  $8 \times 10^{19}$ , and  $8 \times 10^{20}$  n/cm<sup>2</sup> ( $E > 0.1$  MeV).

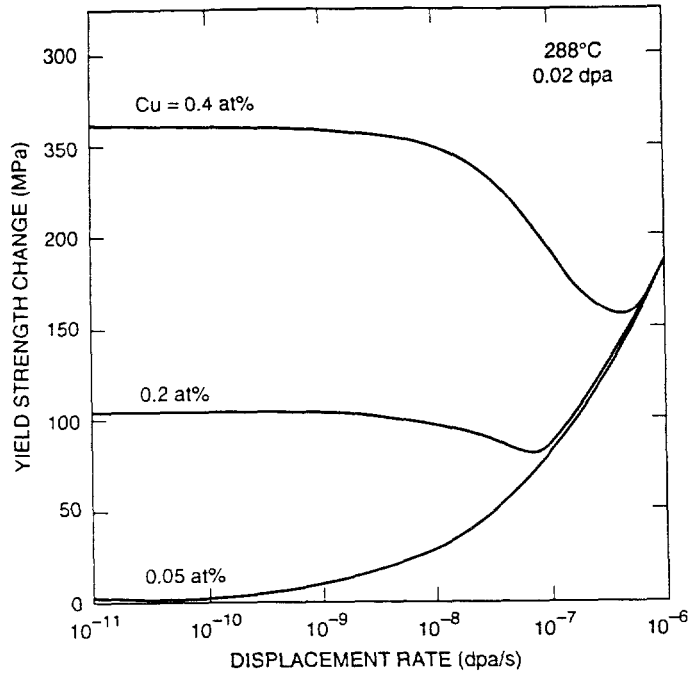


Figure 19. Displacement rate dependence of predicted yield strength change at 0.02 dpa and 288°C.

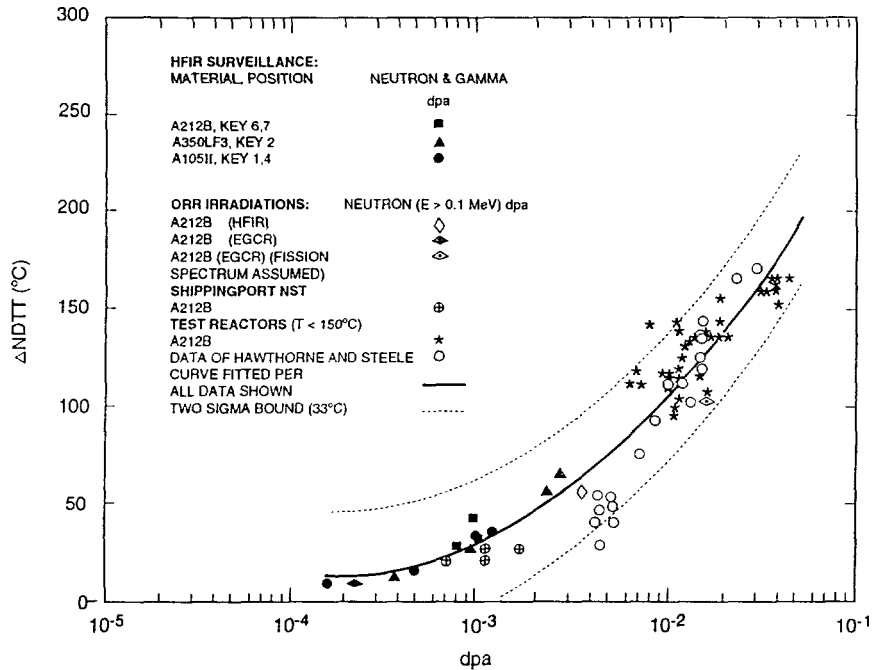


Figure 20. Charpy shift data from High Flux Isotope Reactor surveillance program and other relevant data.

Considerable development work has been required to obtain samples adequate for the required transmission electron microscopy examination and nano-indentation measurements. The formation of a fairly uniform, thin oxide layer can interfere with the nano-indenter measurements at low loads. Several methods were investigated to mitigate this problem, but the final solution required that the lowest load data be discarded. This same oxide film interferes with the TEM examination since the oxide causes a contrast modulation (fringes) in the TEM image that is on the same order (0.5 to 2 nm) as the small defects that are being sought. This may ultimately limit TEM resolution of the smallest point defect clusters, but careful specimen preparation is yielding adequate samples. The initial measurements of hardening and defect cluster size distributions in the irradiated specimens were analyzed using an Orowan strengthening model. The results indicated that the dislocation barrier strength of the radiation-induced defects was about 4, which is consistent with small dislocation loops.

## **9. In-Service Irradiated and Aged Material Evaluations**

**F. M. Haggag, R. K. Nanstad, D. J. Alexander, and P. Arakawa**

Discussed in this chapter are various tasks that evaluate material properties in components of nuclear reactors, including the effects of aging and irradiation. Specific technical activities include (1) procuring and installing a numerically controlled machining center, (2) determining the effects of aging at 343°C (649°F) to 50,000 h on type 308 stainless steel welds, and (3) continuing the studies on stainless steel weld overlay cladding materials. The information reported here includes work from Task 4, Irradiation Effects on Cladding, and Task 14, Additional Requirements for Materials.

### **9.1 Installation of Machining Center in Hot Cell**

Holding fixtures were designed and fabricated to machine three geometries (i.e., miniature flat tensile, CVN, and 1/2 T C(T) compact fracture toughness specimens) using the Computer Numerically Controlled (CNC) machining center (Model VMC-100 of EMCO MAIER, Inc.). K. W. Boling witnessed the machining of these specimen geometries in Columbus, Ohio. Verification of the machine, fixtures, and software was completed, and our purchasing department authorized delivery to ORNL. Delivery of the CNC machine is expected during the summer of 1994. This CNC machine will be modified for remote operation inside hot cell before its installation in the hot cell. A training course at ORNL is also planned during late summer 1994.

### **9.2 Aging of Type 308 Stainless Steel Weld Overlay Cladding**

Thermal aging of type 308 stainless steel welds at 343°C (649°F) for 20,000 h was completed, and tensile, CVN, and fracture toughness specimens were tested. Also, three-wire cladding specimens aged at 288°C (550°F) for 20,000 h were tested. Aging of additional three-wire cladding at 288°C (550°F) or 50,000 h is continuing (completion is expected in July 1996). Results of the completed tests will be discussed in the next semiannual report.

Fourteen specimens were machined from unirradiated single-wire cladding (i.e., types 308 and 309 stainless steel), precracked, and tested to determine dynamic fracture toughness ( $K_{I_d}$ ) values. The twelve irradiated, single-wire cladding, precracked, CVN specimens (i.e., six each of types 308 and 309 stainless steel) were also tested to determine  $K_{I_d}$  values. The specimens and results are being analyzed, and results will be discussed in the next semiannual report.

### **9.3 Aging of Type 308 Stainless Steels Welds**

The stainless steel welds containing 4, 8, or 12% ferrite have completed 50,000 h of aging at 343°C (649°F), and have been removed from the aging furnace. The material has been sent out for fabrication of Charpy and tensile specimens. Tests will be conducted to determine effects of aging on the transition temperature shift and change in ductile (upper-shelf) toughness as well as yield and ultimate strengths. When the tests are completed and results analyzed, a report will be prepared that summarizes the results of all testing and microstructural characterization evaluations on these materials.

## 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 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 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 preparations for moving the material, previously identified and inventoried as correlation monitor material during the previous reporting period, from its current site at the Oak Ridge Y-12 Plant to a controlled-access storage location at ORNL were continued. Detailed planning for the controlled-access location for storage of the remaining correlation monitor materials at ORNL was also initiated. Construction of a concrete slab is planned in mid-1994 as an extension of Building 7026, which will be the future home of the correlation monitor materials. The slab will eventually be covered to shelter the samples from the weather to stop material deterioration. Information is being collected on types of available shelters and price ranges.

Additionally, correlation monitor material from HSST Plate 02 was cut and shipped to A. Kumar at the University of Missouri, Rolla, for his use in studies of irradiation assessment using subsized CVN specimens.

## **11. Special Technical Assistance**

**D. J. Alexander, R. K. Nanstad, F. M. Haggag,  
J. T. Hutton, M. A. Sokolov,  
and E. T. Mannes Schmidt**

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 international codes and standards, of ASME code cases, and of potential deficiencies in proposed supporting technology and data; (2) performing detailed planning and initiating testing of low-alloy steam generator vessel materials with low-temperature postweld heat treatments; (3) initiating the evaluation of using small notched round bar specimens as a fracture specimen for potential surveillance applications; (4) evaluating the use of precracked Charpy specimens and subsize Charpy specimens in assessing material fracture toughness; (5) participating in the Charpy specimen reconstitution round robin coordinated by ASTM by performing the testing of unirradiated specimens; and (6) coordinating the identification, inventorying, shipment, and examination and evaluation of current government-furnished materials and equipment from Materials Engineering Associates-controlled sites to ORNL.

### **11.1 Notched Round-Bar Evaluations**

A section of weld 72W was sent to AEA Technology, Harwell, for their use in testing. Relatively small specimens, 4 and/or 5 mm in diameter, will be fabricated by Harwell and tested at the same temperatures used in the Fifth Irradiation Series for testing of compact specimens.

### **11.2 Evaluation of Nonstandard Charpy Testing**

#### **11.2.1 Initial Evaluation of Precracked CVN Testing**

The computer program used to evaluate CVN specimen tests was upgraded to allow precracked Charpy specimens to be evaluated. The voltage-time trace generated by the instrumented striker is converted to a force-displacement curve. This trace is then analyzed to determine the dynamic fracture toughness  $K_{Id}$ . Several trials were conducted with specimens fabricated from the single-wire stainless steel cladding material. Results are being analyzed and will be described in the next semiannual report.

#### **11.2.2 Initial Evaluation of Subsize CVN Testing**

The main issue in establishing the feasibility of using subsize CVN specimens to determine properties of RPV steels is the correlation of transition temperature and upper-shelf toughness between sub- and full-size specimens. Four types of RPV steels were selected for this study: ASTM A 533, grade B, class 1 plates [one of them after quenching and tempering at 950°C (1742°F)]; specially heat treated steel with A 508, class 2 chemical composition; a Russian forging 15Kh2MFA; and a submerged-arc weld. All of these RPV steels were studied previously at ORNL using standard specimens under different tasks of the HSST and HSSI Programs. The materials were selected to provide a relatively wide range of transition temperatures and upper-shelf energies for standard full-size Charpy specimens as well as a range of yield strengths. To increase the range of properties covered in this study, some steels were examined in the quenched-only or quenched-and-tempered conditions. As a result, the upper-shelf energies of the full-sized specimens varied from 73 to 330 J, the transition temperatures ranged from -46 to +58°C (-51 to 136°F), and the yield strengths varied from 410 to 940 MPa. A total of ten materials/conditions were studied.

The ASTM Method for Notched Bar Impact Testing of Metallic Materials (E-23) allows the use of subsize specimens when the amount of material available does not permit making the standard impact test specimens, but "the results obtained on different sizes of specimens cannot be compared directly." Therefore, the use of subsize specimens recommended by ASTM E-23 requires correlating them with standard specimens. According to ASTM E-23, the length, notch angle, and notch radius for subsize specimens are the same as for full-size specimens, which significantly restricts the possible subsize specimen dimensions. One of the key attractions of subsize specimens for RPV applications is based on using broken halves of full-size surveillance specimens. Five designs of subsize specimens were chosen for the present study (see Figure 21). The type 1 specimen has a length of 25.4 mm, a 5 × 5 mm cross-section, and a 30° notch 0.8 mm deep with a root radius of 0.08 mm. Two type 1 specimens could be machined from one broken full-size Charpy specimen. The type 2 specimen has a length of 25.4 mm, a 3.3 × 3.3 mm cross section, and a 30° notch 0.5 mm deep with a root radius of 0.08 mm. Eight type 2 specimens could be machined from one broken full-size Charpy specimen. One advantage of choosing types 1 and 2 specimens is the accumulated experience of using these subsize specimens in the United States and Japan for fusion materials. The type 3 specimen has a length of 27 mm, a 5 × 5 mm cross section, and a 45° notch 1.0 mm deep with a notch root radius of 0.25 mm. This type of subsize specimen has the same notch profile as the full-size ASTM E-23 standard Charpy specimen. Two type 3 specimens could be machined from one broken full-size Charpy specimen. Experience with this type of subsize specimen has been accumulated in Russia for RPV steels. The type 4 specimen has a length of 26 mm, a 3 × 4 mm cross section, and a 60° notch 1.0 mm deep with a root radius of 0.1 mm. Up to 12 type 4 specimens could be machined from one broken full-size Charpy specimen. Experience with this type of subsize specimen has been accumulated in Europe and Russia for different low-alloy steels including RPV steels. The type 5 specimen has a length of 55 mm, a 5 × 5 mm cross section, and a 45° notch 1.0 mm deep with a root radius of 0.25 mm. This type is the smallest subsize specimen recommended by ASTM E-23. A major disadvantage of this design is that it is not possible to make this type of subsize specimen from a broken full-size Charpy specimen. Nevertheless, specimens of this design were studied for two materials. Table 8 lists the types and properties of the materials.

Subsize specimens have been machined from all of these materials. Tests of each type of subsize specimen from HSST Plate 02 have been completed, and testing of other materials is in progress.

### 11.3 ASTM Reconstituted Round Robin

Reconstituted specimens have been received from six of the participants. The specimens have been sorted and identified in preparation for testing. Specimens have been fabricated with inserts of both 10- and 14-mm length. For each participant the test matrix consists of two insert sizes for each of two materials (i.e., HSST Plate 03 and an LUS weld) tested with each of two strikers (2- and 8-mm radii) at each of two temperatures. The tests of specimens from the current six participants will be tested as a group; then the specimens received by November 1994 from the remaining participants will be tested as a group prior to the end of 1994.

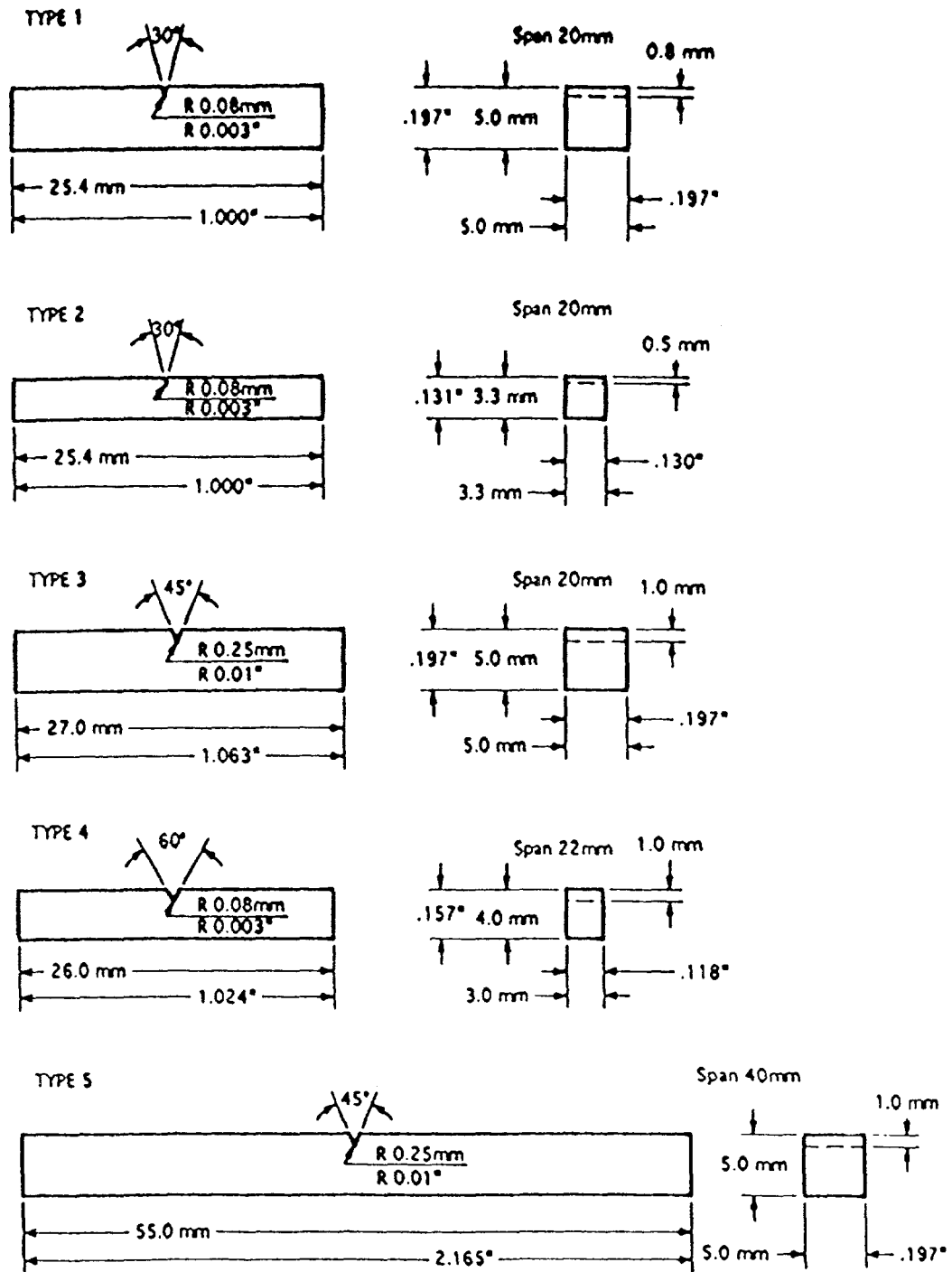


Figure 21. Dimensions of subsized specimens studied.

Table 8. Test matrix and mechanical properties of materials studied

Material	Upper-shelf energy (J)	DBTT <sub>41</sub> (°C)	Yield strength (MPa)	5 x 5 Type 1 U.S./Japan	3.3 x 3.3 Type 2 U.S./Japan	5 x 5 Type 3 Russian	3 x 4 Type 4 German	5 x 5 Type 5 ASTM
A 533 wide plate, LT orientation	330	-43	422	X	X	X	X	-
A 533 wide plate, TL orientation	244	-46	410	X	X	X	X	-
A 508, as-quenched	115	58	634	X	X	X	X	-
A 508, quenched and tempered at 599°C	102	40	697	X	X	X	X	-
A 508, quenched and tempered at 677°C	116	18	605	X	X	X	X	-
A 508, quenched and tempered at 704°C	164	-32	500	X	X	X	X	-
HSST Plate 02, TL orientation	141	0	432	X	X	X	X	X
HSST Plate 014, quenched and tempered at 950°C	73	-34	940	X	X	X	X	-
15Kh2MFA, melt 103672	181	-40	630	X	X	X	X	-
HSSI weld 72W	136	-28	500	X	X	X	X	X

X = 12 to 15 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 that has been irradiated during normal service. This task has been included within 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 was reached some time ago 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 has been very slow movement toward the production of a signed, formal agreement. However, following minor revisions to the original document, the final agreement was signed by JAERI and sent to ORNL for approval. The agreement should be finalized during the next reporting period.

The JPDR was a small boiling-water reactor that began operation in 1963. It operated until 1976, accumulating  $\approx 17,000$  h of operation, of which a little over 14,000 were with the original 45-MW(t) core, and the remaining fraction, late in life, was with an upgraded 90-MW(t) core. The pressure vessel of the JPDR, fabricated from A 302, grade B, modified steel with an internal weld overlay cladding of 304 stainless steel, is approximately 2 m ID and 73 mm thick. It was fabricated from two shell halves joined by longitudinal seam welds located  $180^\circ$  from each other. The rolling direction of the shell plates is parallel to the axis of the vessel. It operated at  $273^\circ\text{C}$  ( $523^\circ\text{F}$ ) and reached a maximum fluence of about  $2.3 \times 10^{18}$  n/cm<sup>2</sup> ( $>1$  MeV). The impurity contents in the base metal are 0.10 to 0.11% copper and 0.010 to 0.017% phosphorus with a nickel content of 0.63 to 0.65%. Impurity contents of the weld metal are 0.11 to 0.14% copper and 0.025 to 0.039% phosphorus with a nickel content of 0.59%.

The current status of the JPDR pressure vessel is that it has been cut into pieces, roughly  $800 \times 800$  mm  $\times$  the original local wall thickness. Full-thickness trepans have been cut from one of the sections originally located at the core beltline and from one of the sections near the upper flange, well away from the beltline. Eight beltline trepans were removed containing the longitudinal fabrication weld, as were eight beltline trepans located completely within the base metal. Nine remotely located trepans were taken containing the longitudinal fabrication weld, as were 14 containing only base metal. JAERI has shipped the irradiated material from the wall of the JPDR that will be examined at ORNL. There it was received, and arrangements were made to move it into the hot cells where it is to be machined and examined. The material received at ORNL consists of 16 full-thickness trepans, each approximately 87 mm in diameter. The trepans contain four types of material: weld metal and base metal, each in both the irradiated condition (from the beltline) and in nominally, thermally aged only condition (from the upper flange). ORNL received four trepans of each material. JAERI has placed all the remaining vessel material in a hot warehouse on-site for long-term storage and currently has no plans to do anything else with it.

The objectives of the JAERI JPDR pressure vessel investigations are to obtain materials property information on the pressure vessel steel actually exposed to in-service irradiation conditions and to help validate the methodology for aging evaluation and life prediction of RPVs. The Japanese research associated with the evaluation of irradiation effects is composed of three parts: examination of material from the JPDR vessel in conjunction with a reevaluation of its exposure conditions, new test reactor irradiations of archival and similar materials, and reevaluation of data from irradiation surveillance and related programs. The focus of the research to be performed by ORNL on the JPDR material is the determination of irradiation-induced damage through the thickness of the vessel in the beltline region and its comparison with the properties and microstructural evaluations of the same material following short, high-rate irradiations or with thermal damage only. This will be done by fabricating fracture and microstructural specimens from the trepans taken from the beltline and from the region remote from the beltline. Parallel determinations of exposure will be made by dosimetry measurements taken on the vessel material itself and by supporting neutron transport calculations.

In anticipation of the implementation of the JPDR testing program, which will include the need to test subsize impact specimens, efforts were initiated to expedite the testing of the subsize specimens. Following the completion of a literature review and analysis of subsize Charpy impact specimen designs, procedures, and data, a test matrix was developed to study the effects of different geometrical parameters on the relationship between subsize and full-size specimens. This matrix includes five types of subsize specimens and ten materials with a wide range of Charpy transition temperatures and upper-shelf energies. All specimens from the materials were machined, and about 70% of the testing of the matrix was completed. Details of this study are described in Chapter 11.

## **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 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 irradiation, annealing, and reirradiation 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**

Sections of two weld metals were supplied by the Russian National Research Center, Kurchatov Institute, for irradiation in a U.S. reactor. Charpy V-notch and round tensile specimens were machined from these welds and placed in HSSI capsule 10.06 for irradiation in the University of Michigan Ford Reactor. The target neutron fluence is  $1 \times 10^{19}$  neutrons/cm<sup>2</sup> (> 1 MeV) at a target irradiation temperature of 288°C (550°F). Some of the Russian specimens, however, were placed in remote parts of the capsule where the irradiation temperature will be close to 270°C (518°F), the operating temperature of some VVER-440 reactors. The welds are identified (Russian designation) as weld 502 (wire SV-10KhMFT), a typical weld metal for VVER-440 reactors, and weld 260-11 (wire SV-12Kh2N2MAA), a typical weld for VVER-1000 reactors. The irradiation should be completed at the end of FY 1994.

### **13.2 JCCCNRS Working Group 3**

Meetings of the JCCCNRS were held in Washington, D.C., and Annapolis, Maryland, in October 1993.

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 coordinated by ORNL and includes testing of two each 0.5 and 1 TC(T) specimens of two materials at 100°C (212°F). 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.

Preliminary results from ORNL and the USNA were presented during the meetings. All the testing and comparative analyses will be completed and reported at the Working Group 3 meeting in 1994.

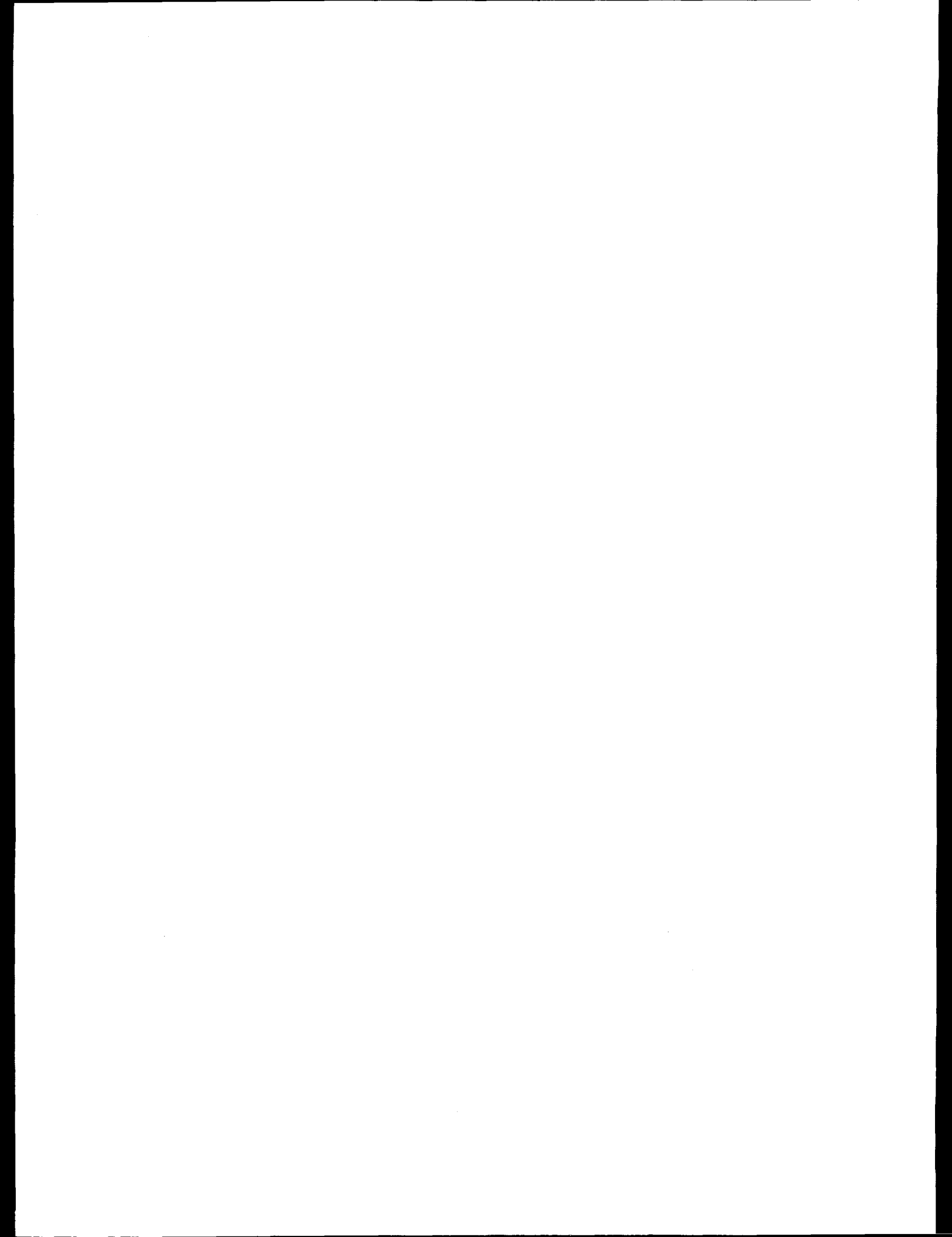
The representatives from the HSSI Program at the meetings were R. K. Nanstad, D. E. McCabe, M. A. Sokolov, S. K. Iskander, W. R. Corwin, and M. K. Miller.

### **13.3 Personnel Interactions**

The HSSI Program is sponsoring the sabbatical of M. A. Sokolov at ORNL. Sokolov's areas of concentration are thermal annealing of irradiated reactor pressure vessel steels and the use of subsized Charpy impact specimens for irradiated studies. The results of his research will be presented within the particular technical tasks of HSSI semiannual progress reports and published technical reports and papers.

## 14. Additional Requirements for Materials

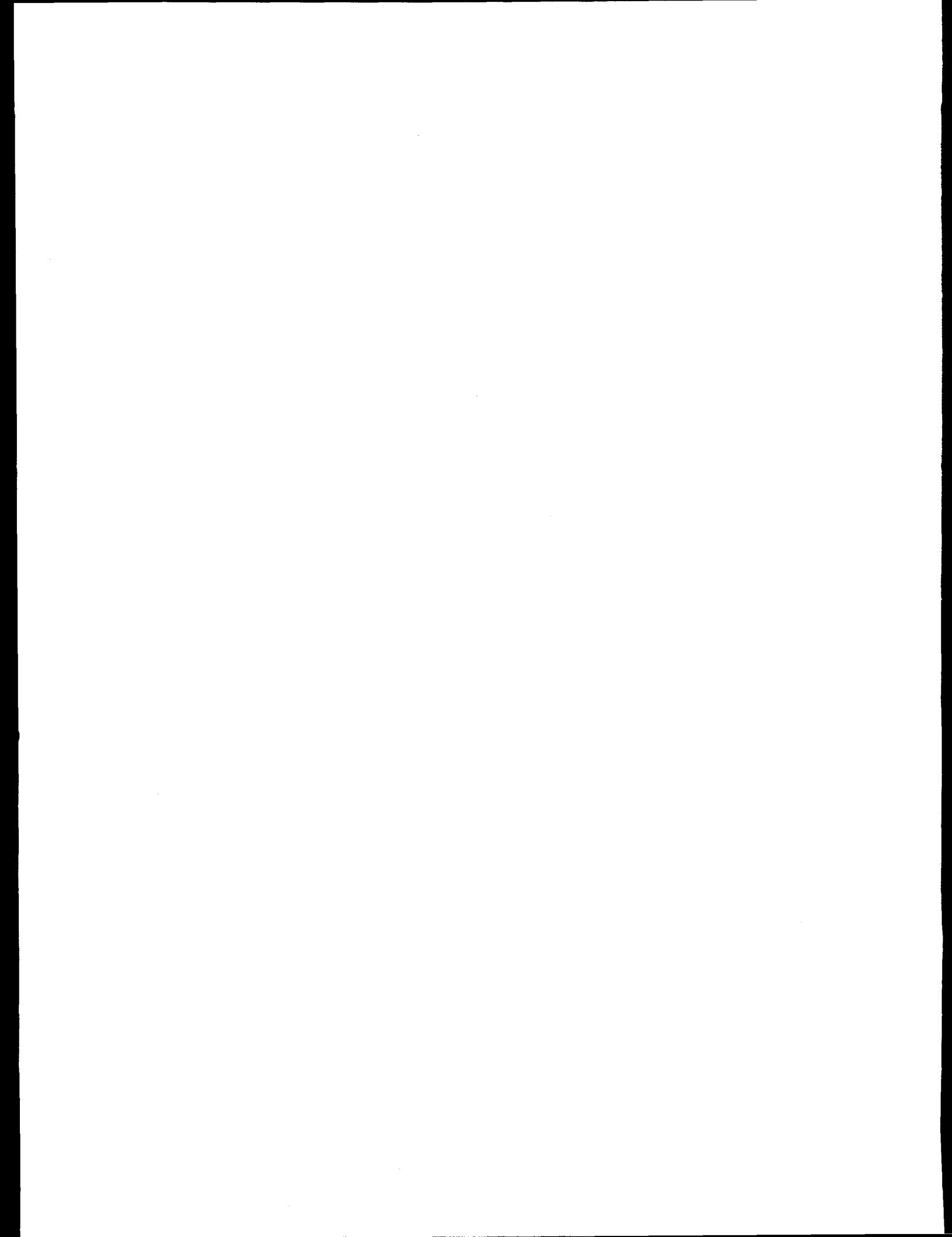
Progress on this task during this reporting period is discussed in Chapter 9.



CONVERSION FACTORS<sup>a</sup>

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 <sub>f</sub>	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 <sup>2</sup>	in.-lb/in. <sup>2</sup>	5.71015
W•m <sup>-3</sup> •K <sup>-1</sup>	Btu/h•ft <sup>2</sup> •°F	1.176110
kg	lb	2.20462
kg/m <sup>3</sup>	lb/in. <sup>3</sup>	3.61273×10 <sup>-5</sup>
mm/N	in./lb <sub>f</sub>	0.175127
T(°F)=1.8(°C)+32		

<sup>a</sup>Multiply SI quantity by given factor to obtain English quantity.



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10. SUPPLEMENTARY NOTES

11. ABSTRACT (200 words or less)

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 September 1993 through March 1994.

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