AIM

Association for Information and Image Management

1100 Wayne Avenue, Suite 1100
Silver Spring, Maryland 20910
301/587-8202

Centimeter

Inches

MANUFACTURED TO AIM STANDARDS
BY APPLIED IMAGE, INC.

1 of 1

DOE/ER/12895--T1

**STUDIES OF LOW TEMPERATURE, LOW FLUX
RADIATION EMBRITTLEMENT OF NUCLEAR
REACTOR STRUCTURAL MATERIALS**

Final Report

DE-FG07-89ER12895

submitted to

Office of Energy Research

Department of Energy

Washington, DC

by

G.R. Odette and G.E. Lucas

Department of Chemical & Nuclear Engineering

University of California, Santa Barbara

June 1, 1993

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

g75

TABLE OF CONTENTS

Section	Page
EXECUTIVE SUMMARY	2
1.0 INTRODUCTION.....	7
2.0 OVERALL WORK SCOPE.....	9
2.1 Task I. MTR Irradiation and Mechanical Property Evaluation.....	9
2.2 Task II. Studies of Shippingport and HFIR Irradiated Alloys.....	11
3.0 RESULTS.....	14
3.1 Initial Piggy back Study	14
3.2 Preparation of Low Flux/Temperature Experiment.....	23
3.2.1 Materials.....	23
3.2.2 Irradiation Capsules and Conditions	30
3.2.3 Post Irradiation Testing.....	31
3.3 Summary of Results.....	34
REFERENCES.....	37

EXECUTIVE SUMMARY

This research was motivated by recent unanticipated results from the High Flux Isotope Reactor (HFIR) surveillance program suggesting that low fluxes (and/or thermal neutrons) may significantly accelerate embrittlement for a range of out of vessel reactor components. Low temperature, low flux embrittlement may also be an issue in several advanced, defense and research reactor systems. Prior to this study there was very little data on the effects of either metallurgical (e.g. composition and processing history) or environmental (e.g. flux, fluence, temperature and spectrum) variables pertinent to this temperature regime. Experience shows that embrittlement of pressure vessel steels at 288°C is mediated by the combined effects of a lengthy list of such variables. Hence, the objective of this program was to: 1) develop a relevant data base on low temperature embrittlement; and 2) to analyze it in the context of physically-based embrittlement models.

Substantial progress was made in addressing these issues based on a "piggyback" experiment carried out in collaboration with ORNL. This experiment involved irradiation of a set of 33 steels in the form of 3 mm diameter, 0.5 mm thick hardness discs representing controlled variations in Cu (0 to 0.8%), Ni (0 to 1.25%) plus one P and one Si variant for up to 3 heat treatments. The irradiations were carried out at temperatures of 65, 121, 163, 204, and 288°C to fluences of about 7×10^{17} n/cm² at fluxes of 9×10^{11} n/cm²-s. The controlled variable matrix was supplemented by tensile specimens for 7 commercial steels included by ORNL and tested by UCSB. The significant results can be summarized as follows:

- Significant hardening was observed ranging from about 10 to 40 kg/mm².
- At temperatures of 163°C and above the hardening increased with Cu and Ni and was relatively insensitive to temperature.

- Hardening was generally lower and insensitive to composition at 121°C.
- The tensile yield stress also appeared to be insensitive to Cu and Ni variations at 121 but not at 288°C. However, several different temperature dependencies of the tensile yield stress (e.g., increases, decreases and approximately no changes with temperature) were observed over the entire range of temperatures. The ultimate tensile strength generally decreased with decreasing temperature consistent with the hardness trends.

These results were interpreted to indicate that the relatively athermal (for $T \geq 121^\circ\text{C}$) component of hardening attributed to copper rich precipitates decreased, or was eliminated, at 121°C and below. The hardening in low copper steels at all temperatures and in all the steels at the higher temperatures was attributed to so-called matrix hardening from such features as nanovoids or small interstitial loops. Further, differences between the temperature dependencies suggest that yield stress increases are more sensitive to ultrafinescale matrix damage (and perhaps very small precipitates) than either hardness or ultimate tensile strength. The latter are measures of the flow stress after appreciable strain (e.g. about 7% for hardness). Hence, we hypothesize that this higher sensitivity of the yield stress is due to shearable defects that are destroyed as dislocation obstacles at after a few percent strain.

We have confirmed the cut-off of copper precipitation at the lowest temperature based on small angle neutron scattering (SANS) measurements. A significant population of copper rich precipitates was observed in a 0.4 C/0.6 Ni steel irradiated at 288°C; however, no features were observed in the same alloy irradiated at 121°C. The 288°C irradiation produced a hardening of 22 kg/mm² versus 12 kg/mm² for the lower temperature. The extra hardening at 288°C is consistent with the observed size, number density and volume fraction of the precipitates.

In addition we have performed a post-irradiation annealing study on one of the copper bearing alloys. Complete recovery was observed after 35 hours of annealing at 343°C for materials irradiated below 163°C. This behavior is also consistent with the picture of predominantly matrix defect hardening at the low irradiation temperatures.

In spite of the importance of these results, they are limited and, for example, cannot be extrapolated to much higher fluences or lower flux regimes. Hence, we made substantial progress in producing the most extensive matrix of steel alloys ever assembled for an embrittlement study. Overall the matrix represents a total of 82 alloy conditions. This matrix has permitted us to begin to evaluate not only the the separate but also combined effects of Cu and Ni and also P and Mn, as well as the influence of elements such as Cr, N, C and others. Design of this matrix was guided by our extensive studies of 288°C pressure vessel embrittlement.

Mechanical property and microstructural specimens (about 3500 miniature tensiles, and 2700 discs , 300 SANS and 100 coupons and FIM specimens) have been fabricated. A subset of these have been irradiated in the University of Michigan Ford Reactor at a flux of $2 \times 10^{11} \text{n/cm}^2\text{-s}$ to fluences of 0.2 and $0.5 \times 10^{18} \text{n/cm}^2$ and post-irradiation tensile testing has been performed on some of these specimens. Data from these specimens suggest a relatively weak dependence of hardening on metallurgical variables; when combined with other existing low-temperature irradiation data, the data also suggest a relatively weak dependence on flux down to $2 \times 10^{11} \text{n/cm}^2\text{-s}$; these trends, however, also point up the importance of additional lower flux irradiations to be achieved in the follow-on irradiations proposed here. One full set of specimens has been irradiated at $7 \times 10^{11} \text{n/cm}^2\text{-s}$ to a fluence of $3 \times 10^{18} \text{n/cm}^2$. Three more full sets of specimens have been left in reactor to receive irradiation at a flux level of $\sim 0.02 \times 10^{12} \text{n/cm}^2\text{-s}$ to fluences of 2 and $5 \times 10^{17} \text{n/cm}^2$ and at a flux level of $2 \times 10^{11} \text{n/cm}^2\text{-s}$

to a fluence of $3 \times 10^{18} \text{n/cm}^2$. Under a new program, we will continue these irradiations and complete several additional irradiations as described below.

Since, practical MTR irradiations cannot access the very lowest flux regime we have devised an approach for using irradiated materials from both the HFIR surveillance program and the Shippingport shield tank in a novel combination of post-irradiation annealing and re-irradiation embrittlement experiments. Among other objectives, this task seeks to identify means of reliably establishing the pre-irradiated condition of steels sampled from actual reactor components both in terms of their mechanical properties and relative radiation sensitivity. In the long term, this effort could establish procedures to mitigate problems associated with lack of archival material that often confound interpretation of data from component sampling programs.

As part of the overall effort, we upgraded our automated hardness testing capability and designed and built an automated miniature tensile testing system.

It should be noted that an enormous effort has gone into: designing the overall experiment; acquiring the base alloys; developing and carrying out heat treatments to produce the desired unirradiated properties and microstructures; fabricating specimens and verifying test techniques; designing the capsules and planning of the irradiation procedures; and characterizing and annealing the HFIR and Shippingport samples. To this end we thankfully acknowledge the enormous contributions of Dr. Roger Stoller of ORNL who spent a sabbatical year at UCS² and Dr. Will Phythian of the UK Harwell Laboratory.

Data generated to date from the Michigan irradiations have shown that strengthening is approximately proportional to the square root of fluence. Comparison to comparable data in the literature suggests that at the low fluences examined, there is little effect of irradiation flux above about $2 \times 10^{11} \text{n/cm}^2\text{-s}$. Within the limited data

obtained so far, there appears to be a relatively weak dependence on nickel, copper, manganese and phosphorous content. These data have helped to define a final irradiation matrix to address the issue of low flux, low temperature embrittlement in a follow-on program.

1.0 INTRODUCTION

Safe and reliable operation of nuclear power plants requires accurate assessment of the properties of structural materials over their entire operating lifetime. Materials often undergo time-dependent changes in important properties as a consequence of service in the hostile environment of a reactor system. Significant degradation of properties must be predicted in advance so that safe design can be ensured and, if necessary, appropriate remedial action can be taken (*i.e.* repair or replacement). Accurate property forecasting is particularly important to regulatory issues regarding plant life extension (PLEX) for existing plants and will play a key role in the development of advanced and more inherently safe reactors.

Recent data suggest that structural steels may experience significant embrittlement (*i.e.* transition temperature shifts $>200^{\circ}\text{C}$) even at relatively low fluences at low service irradiation temperatures (about 50°C). Specifically, data from the High Flux Isotope Reactor (HFIR) pressure vessel evaluation program suggest that more embrittlement per unit fluence occurs at low fluxes than at high fluxes [1,2,3]. Similar flux effects have been observed not only at intermediate temperatures [4] and but also at higher temperatures characteristic of pressure vessel conditions [5], although the magnitude and the mechanisms mediating the flux effect probably vary with temperature. Thus a range of components of both existing and future reactors -- such as reactor support structures -- which are exposed to relatively low neutron fluxes at low temperatures, may experience significant and previously unanticipated levels of embrittlement. Consequently, low flux/ low temperature embrittlement is a phenomenon with significant generic potential importance to a wide variety of current and future nuclear power plants.

Since embrittlement under these conditions was not anticipated to be a problem affecting the life of current power plants, there are no surveillance programs for these structures. Consequently, assessments of the degree of embrittlement must be made by tests on existing plant materials and/or extrapolations of data obtained in materials test reactors (MTR's). There is currently nearly a complete void in both the data base and understanding that is required to responsibly and reliably predict low-temperature, low-flux embrittlement. The role of thermal neutrons, if any, is also unknown and must be assessed.

This program has been directed at acquiring a better physical understanding of the governing phenomena. This understanding will ultimately be used in developing and calibrating against experimental data physical models that describe the dominant controlling processes based on this understanding. These models will provide a much more reliable basis to interpolate and extrapolate the engineering data than estimates based on purely empirical "trend curve" approaches. We have previously shown that such models provide a much improved capability for predicting higher temperature ($\sim 300^{\circ}\text{C}$) neutron irradiation embrittlement of reactor pressure vessel steels [6-9].

This program has focussed on a significant experimental effort to develop an adequate data base to permit model development and calibration. Flux effects have been addressed by what we believe to be an innovative combination of both controlled MTR irradiations and by evaluation of samples extracted from the Shippingport reactor shield tank and samples from the High Flux Isotope Reactor (HFIR) surveillance program. Specimen preparation, irradiations and testing were carried out in coordination and collaboration with related programs at ORNL, ANL, Sandia National Laboratories, and AEA Technologies, Harwell.

2.0 OVERALL WORK SCOPE

There are several existing research programs which have components pertinent to the issue of low flux/low temperature embrittlement; in particular, examination of the Shippingport shield tank which has been exposed to low flux and relatively low temperature is being performed by ANL [10], and evaluation of low temperature embrittlement in A508 and A533B steels in support of the HTGR program is currently being conducted by ORNL [11]. However, these programs are not specifically directed at the broader issue of low flux/low temperature embrittlement in a range of structural steels. Hence, we coordinated our effort with these programs so that our investigations were complementary to these, and we focused on a set of materials which expand the data base developed in these programs. In particular, we have investigated embrittlement phenomena in steels that are similar to those used in support structures.

2.1 Task I. MTR Irradiation and Mechanical Property Evaluation

Our program was set up to perform single variable (*e.g.* split melt) scoping studies of important compositional (*e.g.* copper, nickel, manganese, carbon, nitrogen...), microstructural (*e.g.* ferrite, bainite, and grain size...) and environmental variables (temperature, flux, and spectrum).

Irradiation of these steels was planned over a range of fluxes (ϕ) and overlapping fluences (ϕt) ranging from low values of $\phi \sim 2 \times 10^{10}$ n/cm²-s up to $\phi t \sim 5 \times 10^{17}$ n/cm² to high values of 5×10^{14} n/cm²-s up to 7×10^{18} n/cm². Miniaturized tensile specimens and/or hardness specimens were planned for each material/irradiation condition to provide a measure of the irradiation-induced change in the yield stress $\Delta\sigma_y$. We have previously demonstrated that transition temperature shifts (ΔT_{41}) and upper shelf energy changes (ΔUSE) are related to $\Delta\sigma_y$ for higher temperature

irradiations (288°C) on reactor pressure vessel steels [12], and preliminary evidence suggests that a similar relation is true for lower temperature [2]. Hence, measurements of $\Delta\sigma_y$ can be used as an indication of the sensitivity of the various steels to embrittlement. As described later, we developed the capability to perform a large number of such tests using automated systems that we have developed in both this and other programs.

The approach to assessing low flux effects on low temperature embrittlement based on MTR irradiations is schematically illustrated in Figure 2.1. Here, we have taken some current support structure service conditions as the nominal parametric regime of interest. However, the regimes for other components are not too dissimilar and are addressed in the research. For support structures, the lowest flux/ highest fluence combination (presumably corresponding to the largest shift) is $\phi=3\times10^9\text{n/cm}^2\text{-s}$ and $\phi t=3\times10^{18}\text{n/cm}^2$ [13]. The planned MTR irradiations provide data at flux levels about a factor of 7 to 3×10^5 higher at fluences about a factor of 6 below to 2 above these conditions. These estimates of fluxes and fluences are based on displacement-per-atom (dpa) equivalents, since dpa is the most appropriate exposure parameter for treating spectral effects in low temperature embrittlement. Hence, results must be extrapolated to low flux (service) conditions. Figure 2.1a shows schematically how such an extrapolation can be based. Data from the planned irradiations can provide estimates of the fluence dependence of hardening/embrittlement (e.g., a square root dependence) at several flux levels, and the observed trends can be used to estimate fluence dependence at the lower flux levels (shown as the bold line in Fig. 2.1a). This estimate is based on extrapolations shown in the cross plots in Fig. 2.1b; here the fluence required to produce some specified level of hardening (embrittlement) at a flux level ϕ is normalized by the same fluence obtained under flux levels of $3\times10^{12}\text{ n/cm}^2\text{-s}$ and plotted against flux. The several curves shown in Fig. 2.1b are for steels with

Figure 2.1 a) Schematic of experimentally deduced hardening ($\Delta\sigma_y$) versus fluence (dpa) as a function of flux; extrapolation to support structure regime is shown as the bold line; b) Schematic cross plots of the fluence required to produce $\Delta\sigma_y$ normalized to $\phi = 3 \times 10^{12} \text{ n/cm}^2$; c) Schematic cross plot of $\Delta\sigma_y$ versus metallurgical variables (MV).

different composition/heat treatments. Extrapolation of these data to low flux (service) conditions can provide an estimate of the effects of these variables on support structure embrittlement as shown in Fig. 2.1c. Of course, the extrapolations should be tested. However, extension of the test reactor data base to lower fluxes or higher fluences would require longer irradiation times; these times are prohibitively long (by a factor of about 40) if actual service conditions were to be approximated. An alternate approach is to test steels that have already been irradiated under low temperature and low flux conditions for extended periods of time, and this is outlined in the next Task.

2.2 Task II. Studies of Shippingport and HFIR Irradiated Alloys

In this task we examine steels already irradiated under low flux low temperature conditions. Ideally, one would like to have both unirradiated and irradiated properties on the same heat of steel to avoid the effects of metallurgical variables confounding the results; one would also like to have archival material both to verify the unirradiated properties and to use in higher flux irradiation experiments. Unfortunately, there is currently only one case where this is known to be possible: namely, for two steels (A212 and A350) irradiated in the HFIR surveillance program. The fluxes and fluences in these HFIR irradiations were about 10 times below the maximum flux/fluence regime for support structures. Hence, irradiation of these two alloys in the test matrix proposed in Task I extend the flux regime to the lower limit of interest for support structures. This is illustrated schematically in Figure 2.2. However, the fluences are lower than maximum end-of-life support structure fluences at the low-to-intermediate fluxes.

The only apparent source of data closer to actual support structure service conditions is samples from components that have experienced actual service for long periods of time. The NRC supported a program at ANL to examine such samples from

Figure 2.2. Schematic illustration of the variation of fluence to achieve a specific level of embrittlement as a function of flux, showing how MTR and HFIR/ORR data can be used to verify the extrapolation of flux dependence to the low flux regime.

the Shippingport reactor neutron shield tank [10]. The usefulness of data from such studies is limited, however, because:

- 1) there are considerable uncertainties in the fluxes and fluences experienced by various parts of the shield tank;
- 2) neither archival materials nor unirradiated properties are available for the specific plates and welds used in the Shippingport shield tank; hence, it is not possible to evaluate explicitly the changes induced by low flux irradiation (shield tank components with low fluences will be used to estimate the initial properties; however, heat-to-heat variations in the initial properties may confound precise assessment of irradiation induced changes);
- 3) the radiation sensitivities of the Shippingport plates and welds are not known relative to other steels in service.

Hence, while the Shippingport data are useful, they do not fully address low flux/fluence embrittlement issues.

The innovative experiments in our program are directed at mitigating some of the problems associated with the Shippingport studies. In addition to being useful to the objectives of this study, the technique that will be demonstrated here could be very useful to other component sampling studies. The experiments involve post-irradiation-annealing (PIA) to: 1) recover the initial properties (addressing limitation 2 above) and, 2) *possibly* recover the initial metallurgical state of the Shippingport steels. Reirradiation embrittlement (RIE) studies will be used to test the *hypothesis* that the initial metallurgical state can be recovered by PIA. If this hypothesis is correct, comparisons of the Shippingport steels to other steels irradiated in Task I can be used to rank their relative radiation sensitivity (addressing limitation 3 above). The experimental sequence is as follows:

- 1) A subset of the HFIR A212 and A350 surveillance specimens are annealed to recover their initial properties (which are known);
- 2) Both archival (unirradiated) and PIA (recovered) HFIR alloys are irradiated over the range of fluences and fluxes shown in Task 1.
- 3) Similarity in the irradiation behavior of the unirradiated and PIA steels would suggest that PIA recovers the aspects of the initial metallurgical state critical to irradiation response at low fluxes.
- 4) A subset of other steels initially irradiated at intermediate and high fluxes will also be subject to the same type of assessment to provide further evidence about the maintenance of similar radiation sensitivity following cycles of irradiation-PIA-reirradiation.
- 5) A similar sequence of irradiation, PIA, RIE which can be applied to samples of the Shippingport steels. Assuming the above hypothesis is verified, these data would provide estimates of the unirradiated properties of the Shippingport steels and rank their relative radiation sensitivity.

Figure 2.3 schematically illustrates this cycle of irradiations, PIA, and reirradiations. In addition to providing mitigation of the issues noted above, the PIA/RIE studies provide direct data on similarities and differences in the irradiation and RIE behavior of steels of interest and provide data relevant to assessing a PIA approach to recovery of embrittled support structures.

Because quantities of these materials are be limited, and because they are radioactive, we perform microhardness tests to interrogate their mechanical properties. Specimens for microhardness testing can be made relatively easily and require small volumes of material. We have previously demonstrated that changes in Vickers

Figure 2.3. Schematic illustration of the proposed irradiation, PIA, RIE experiments. P_A = property (DPH) in the annealed condition, P_I = property in the irradiated condition, P_R = property in the re-irradiated condition, P_O = initial unirradiated property.

microhardness (ΔDPH) are proportional to $\Delta\sigma_y$ and hence can be related to ΔT_{41} and ΔUSE as indicated earlier [5,9].

3.0 RESULTS

We assembled a very large irradiation experiment that consists of 82 materials and 4 specimen types for irradiation at 10 conditions. As noted previously, this metallurgical matrix has been expanded considerably over the one described in the original proposal, as we have taken advantage of a collaboration with AEA Technologies/Harwell to procure and characterize this large number of alloys. The irradiation capsules, in-reactor fixturing to hold these capsules, and equipment to perform post-irradiation testing has been built and tested. A subset of the alloys have been irradiated at a flux of $2 \times 10^{11} \text{ n/cm}^2\text{-s}$ to fluences of 0.2 and $0.5 \times 10^{18} \text{ n/cm}^2$ and post-irradiation tensile testing has been performed on each of the materials. In addition, four full sets of specimens have either been irradiated or have been left in reactor for continued irradiation under a follow-on program, and capsules have been prepared to complete the irradiation matrix under the follow-on program. Finally, we performed a piggy-back irradiation experiment to assess the role of compositional variables on irradiation hardening as a function of irradiation temperature down to relatively low levels. Specific accomplishments are described in more detail below.

3.1 Initial Piggyback Study

We performed an initial study to get some preliminary estimates of the effect of irradiation temperature on hardening for material types similar to (and in some cases

identical to) the ones planned for the comprehensive irradiation matrix. We examined two sets of materials in this study. The first was a set of thirty-three A533B-type model alloys, produced from split melts with controlled compositions and heat treatments. These are described in Table 3.1.1. The primary metallurgical variants were copper (up to 5 levels at about 0.8% nickel), nickel (up to 4 levels at about 0.4% copper) and both silicon and phosphorous (2 levels at about 0.4% copper and 0.8% nickel); all compositions were included in two heat treatment conditions (tempered and tempered plus stress relieved); a subset was included in a third heat treatment condition (tempered plus two stress reliefs). The heat treatments were intended to vary the amount of copper in solution prior to irradiation. These alloys were irradiated in the form of 3mm diameter, 0.5mm thick hardness discs. The use of hardness specimens was imposed by space constraints: i.e., irradiations were performed as "piggyback" irradiations in an ORNL experiment, and available volume was limited to one Charpy-V-notch specimen equivalent.

The second was a set of seven commercial steels, which had a range (uncontrolled) of compositions and heat treatments. These are described in Table 3.1.2. They were irradiated in the form of miniature tensile specimens (SS3's, which are 25.4 mm long by ~ 5mm wide by ~ 0.76 mm thick) along with the hardness discs. Irradiations were carried out at flux of about 9×10^{11} n/cm²/s to a fluence of about 7×10^{17} n/cm² ($E > 1$ MeV) at temperatures of 288, 204, 163, 121 and 65°C in the SUNY Buffalo reactor. Fluence and temperature uncertainties are estimated to be $\pm 20\%$ and $\pm 10^\circ\text{C}$, respectively.

Following irradiation Vickers diamond pyramid microhardness (DPH) measurements (500 g load) were obtained from the disc specimens using the semiautomated microhardness tester described later (section 3.2.3). A minimum of 5 indents was used to obtain an average measurement. Changes in diamond pyramid

Table 3.1.1
Compositions of the A533B-type Model Alloys (Wt. %)

Alloy HT ^b Desig.	Composition Versus Base ^a			HT ^b	Alloy Desig.	Composition Versus Base ^a			HT ^b	Alloy Desig.	Composition Versus Base ^a			HT ^b
	Cu	Ni	Other			Cu	Ni	Other			Cu	Ni	Other	
LA	0.400	0.001		SR	LM	0.400	0.001		T	LY	0.400	0.001		2SR
LB	0.400	0.175		SR	LN	0.400	0.175		T	LZ	0.410	0.860		2SR
LC	0.410	0.860		SR	LO	0.410	0.860		T	PA	0.380	1.250		2SR
LD	0.380	1.250		SR	LP	0.380	1.250		T	PB	0.420	0.850	0.025 P	2SR
LE	0.420	0.850	0.025 P	SR	LQ	0.420	0.850	0.025 P	T	PC	0.420	0.805	0.73 Si	2SR
LF	0.420	0.805	0.73 Si	SR	LR	0.420	0.805	0.73 Si	T	PD	0.005	0.740		2SR
LG	0.005	0.740		SR	LS	0.005	0.740		T	PE	0.200	0.740		2SR
LH	0.110	0.740		SR	LT	0.110	0.740		T	PF	0.800	0.810		2SR
LI	0.200	0.740		SR	LU	0.200	0.740		T	PG	0.210	1.100		2SR
LJ	0.420	0.805		SR	LV	0.420	0.805		T					
LK	0.800	0.810		SR	LW	0.800	0.810		T					
LL	0.210	1.100		SR	LX	0.210	1.100		T					

^a Nominal base composition: 0.15 C, 1.37 Mn, .22 Si, .005 P, .005 S, .06 Cr, .55 Mo

^b Heat Treatments: T (Tempered) : Austenitize 900°C, 1h; Air cool; Temper 664°C, 4h, Air Cool
SR (Stress Relieved): T + Air cool; Stress relieve 600 °C, 40h; Furnace Cool to 300 °C; Air Cool
2SR (Twice Stress Relieved): SR + Stress relieve 625 °C, 8h; Furnace cool to 300 °C; Air cool

Table 3.1.2
Compositions of the Commercial Steels

Alloy	Designation	Composition (wt %)				Ni	Mo	Cu	Si	Cr
		C	Mn	P	S					
A	HFIR A212B	0.26	0.85	0.006	0.04	0.2	0.02	0.15	0.29	0.075
C	HFIR A350	0.17	0.5	0.007	0.01	3.2	0.03	0.10	0.27	0.08
GF	A533B, Cl 1	0.22	1.35	0.007	0.016	0.64	0.53	0.07	0.24	0.07
HF	A533B, Cl 1	0.20	1.47	0.005	0.004	0.58	0.48	0.14	0.23	0.14
NW	HFIR Nozzle Weld	0.045	0.92	0.008	0.04	0.10	0.52	0.05	0.55	0.04
SW	HFIR Seam Weld	0.058	1.5	0.015	0.02	0.01	0.48	0.18	0.54	0.04
73W	HSST SA Weld	0.098	1.56	0.005	0.005	0.60	0.58	0.31	0.45	0.250

hardness (ΔDPH) were determined as the difference between the average irradiated and corresponding unirradiated conditions. The average standard deviation in individual hardness measurements was about 5 kg/mm² (DPH units). However, differences between different sets of hardness measurements (ie, different groupings of indents) in the same or equivalent specimens were larger in some cases. Miniature tensile specimens were tested at a strain rate of $3 \times 10^{-4} \text{ s}^{-1}$ in the Instron screw driven load frame described below, from which 0.2% offset yield stresses (σ_y) and ultimate tensile strengths (UTS) were obtained. Uniform and total elongations were also recorded but are not considered here. Again changes in yield ($\Delta\sigma_y$) and UTS (ΔUTS) were determined from differences in the irradiated and unirradiated material. The average standard deviation in the yield and ultimate strength data was about 35 MPa. Changes in yield stress and hardness have been demonstrated to track transition temperature shifts in pressure vessel steels irradiated at temperatures in the neighborhood of 290°C.

One relatively large uncertainty associated with microhardness change measurements derives from various inherent physical sources of "local" variability in both the baseline and irradiated samples. The low fluences in this experiment exaggerate this problem in at least two ways: 1) the hardness changes are relatively small (only on-the-order of 10% of the baseline hardness), and 2) due to the nature of the basic mechanisms, radiation damage itself is likely to be more variable in this regime. A variety of additional sources of measurement variance and bias also contribute to uncertainties and scatter. While these problems cannot be eliminated, they can be mitigated by averaging like data sets. Such averaging establishes smoothed data trends and filters fluctuations, real or otherwise, that are below the reliable resolution limit of the measurement technique. The specific averaging procedures used in this

study are noted below. As a corollary, attempts to interpret behavior of a single or small set of data may be highly misleading.

Typical examples of temperature dependence of hardening are shown in Figure 3.1.1 for twice stress relieved alloys with high and low copper contents (at about 0.8% nickel) and with high and low nickel contents (at about 0.4% copper). Hardening between 288°C and 163°C is relatively insensitive to irradiation temperature, although some other data (not shown here) exhibit a modest decrease in hardening with increasing temperature in this range. However, between 163 and 121°C, hardening decreases with decreasing irradiation temperature, particularly in the copper bearing steels. The data show a clear effect of copper at the three highest temperatures. However, the effect of copper decreases at the lowest temperature and the effect of nickel variations cannot be resolved within the data scatter. Hence, data averaging is used to extract general data trends for the metallurgical variables.

Figure 3.1.2 shows the effect of heat treatment on hardening, where the hardness increases are averaged for all alloys with copper contents of 0.4% and 0.8%. The differences are minimal. Hence, examining compositional dependence of hardening, data were averaged for all three heat treatments. Figure 3.1.3 shows these compositional dependences. The effects of phosphorous (Figure 3.1.3a) and silicon (Figure 3.1.3b) variations appear to be minimal at both the high and low temperatures, although some decrease in hardening with increasing silicon content might be suggested. At temperatures of 163°C and above, the average hardening increases with increasing copper (Figure 3.1.3c), but this copper sensitivity is not apparent at 121°C. There is some suggestion that hardness may increase slightly with nickel content (Figure 3.1.3d) at the three highest temperatures; but, again, a nickel sensitivity is not apparent at the lowest temperature.

Figure 3.1.1 Representative data showing changes in hardness as a function of irradiation temperature for three of the tempered and twice stress relieved materials.

Figure 3.1.2 Variation of hardening heat treatment. Data are averaged for all alloys with 0.4% Cu and 0.8% Ni.

Figure 3.1.3 Variation of hardening with composition: a) phosphorous, b) silicon, c) copper, and d) nickel. Data were averaged for all heat treatments.

(Figure 3.1.3d) at the three highest temperatures; but, again, a nickel sensitivity is not apparent at the lowest temperature.

Examples of the temperature dependence of the yield stress change data are shown in Figure 3.1.4a for a range of copper and nickel contents. A variety of trends are observed, including: $\Delta\sigma_y$ approximately constant with temperature (0.31 Cu, 0.60 Ni), $\Delta\sigma_y$ monotonically increasing with decreasing temperature (0.07 Cu, 0.64 Ni), and $\Delta\sigma_y$ peaked at about 200°C (0.18 Cu, 0.01 Ni). With the exception of the latter case, however, significant reductions in $\Delta\sigma_y$ at the lowest temperature are not observed (including data not shown). Thus the effects of irradiation on $\Delta\sigma_y$ and ΔDPH appear to differ at low temperatures. Note this is clearly not a controlled single variable comparison since tensile and hardness tests were not carried out on the same set of alloys. However, Figure 3.1.4b shows the ultimate tensile strength increases for the same copper-bearing steels also tend to decrease at the lower temperatures. This behavior was also exhibited in the other copper-bearing alloys not shown. Hence, the temperature dependence of ΔUTS and ΔDPH appear to be similar. These trends are also reflected in Figure 3.1.5 where the $\Delta\sigma_y$ -to- ΔUTS ratio -- averaged for the seven alloys -- is shown to increase from about 1.25 to 2.25 with decreasing temperature, suggesting that the features induced by low temperature irradiations have a stronger influence on $\Delta\sigma_y$ than on ΔUTS . Moreover, the close correspondence of the temperature variations of ΔDPH and ΔUTS data is probably associated with the fact that both are measures of strength at significant plastic strain (e.g., hardness corresponds to a effective flow stress at about 7%).

Figure 3.1.6 shows that the $\Delta\sigma_y$ data for the seven steels at 288°C are consistent with normal observations of hardening increases with copper (i.e., levels of 0.07, 0.14, and 0.31 at about 0.6% Ni) and nickel (i.e., levels of 0.01, 0.2 and 0.58 at about 0.14 to 0.18% Cu). However, a similar effect of copper and nickel is not

(a)

(b)

Figure 3.1.4 Examples of the variation of a) yield strength changes and b) changes in UTS with irradiation temperature for commercial steels containing a range of copper and nickel contents.

Figure 3.1.5 Variation in the average ratio of $\Delta\sigma_y$ to ΔUTS as a function of irradiation temperature for all seven commercial alloys.

Figure 3.1.6 Comparison of yield strength change at the high and low irradiation temperatures of all 7 commercial steels. Boxes highlight the copper and nickel variation for nominally comparable steels.

Examples of the temperature dependence of the yield stress change data are shown in Figure 3.1.4a for a range of copper and nickel contents. A variety of trends are observed, including: $\Delta\sigma_y$ approximately constant with temperature (0.31 Cu, 0.60 Ni), $\Delta\sigma_y$ monotonically increasing with decreasing temperature (0.07 Cu, 0.64 Ni), and $\Delta\sigma_y$ peaked at about 200°C (0.18 Cu, 0.01 Ni). With the exception of the latter case, however, significant reductions in $\Delta\sigma_y$ at the lowest temperature are not observed (including data not shown). Thus the effects of irradiation on $\Delta\sigma_y$ and ΔDPH appear to differ at low temperatures. Note this is clearly not a controlled single variable comparison since tensile and hardness tests were not carried out on the same set of alloys. However, Figure 3.1.4b shows the ultimate tensile strength increases for the same copper-bearing steels also tend to decrease at the lower temperatures. This behavior was also exhibited in the other copper-bearing alloys not shown. Hence, the temperature dependence of ΔUTS and ΔDPH appear to be similar. These trends are also reflected in Figure 3.1.5 where the $\Delta\sigma_y$ -to- ΔUTS ratio -- averaged for the seven alloys -- is shown to increase from about 1.25 to 2.25 with decreasing temperature, suggesting that the features induced by low temperature irradiations have a stronger influence on $\Delta\sigma_y$ than on ΔUTS . Moreover, the close correspondence of the temperature variations of ΔDPH and ΔUTS data is probably associated with the fact that both are measures of strength at significant plastic strain (e.g., hardness corresponds to a effective flow stress at about 7%).

Figure 3.1.6 shows that the $\Delta\sigma_y$ data for the seven steels at 288°C are consistent with normal observations of hardening increases with copper (i.e., levels of 0.07, 0.14, and 0.31 at about 0.6% Ni) and nickel (i.e., levels of 0.01, 0.2 and 0.58 at about 0.14 to 0.18% Cu). However, a similar effect of copper and nickel is not observed at 121°C. This behavior is consistent with the hardness data for the A533B model steels with controlled variations in copper and nickel content.

However, the 121°C data do indicate some sensitivity to metallurgical variables. Specifically, two welds irradiated at this temperature have appreciably smaller yield stress increases than the other alloys. These steels share a common feature of having the lowest carbon content (~0.05%), possibly suggesting that this is an important variable in low temperature irradiations. These "insensitive" steels also have the lowest nickel contents (0.01 to 0.1%) and one has a low copper content (0.05%); however, other low copper (0.07 to 0.1%) and low nickel (0.2%) steels fall into the "sensitive" group at 121°C, with larger values of $\Delta\sigma_y$. Unfortunately, we cannot draw firm conclusions about compositional effects from these tensile data due to the confounding effects of uncontrolled variables. Evaluation of the role of elements such as carbon and nitrogen will require an experiment involving controlled variations of these elements, which as noted below is part of the matrix we are examining.

The relatively large hardness increase for irradiation temperatures above 163°C (~30 DPH) suggests a major role of irradiation-enhanced copper precipitation even at a modest fluence of about $7 \times 10^{17} \text{ n/cm}^2$. The relatively athermal (temperature insensitive) character of the copper contribution is broadly consistent with previous elevated temperature data for high fluences where copper precipitation is nearly complete (note this athermal behavior may be somewhat more surprising for the low fluences in this study, where precipitation is presumably incomplete). More significantly, however, there appears to be a decrease in the contribution of copper precipitation below 163°C.

The modest magnitude of the nickel sensitivity observed in the hardness data at the higher temperatures is consistent with previous observations that the contribution of this element increases with increasing fluence. However some previous studies, conducted in the range of about 250 to 310°C, suggested that the effect of nickel increases with decreasing temperature. We have postulated that in combination nickel and lower temperature promote hardening contributions from copper-independent

matrix defects. However, an increased effect of nickel with decreasing temperatures is not observed in this low fluence experiment. Indeed, like copper the effect of nickel appears to decline below 163°C reaching negligible levels at 121°C. The coupling of the copper and nickel trends may indicate that, rather than being independent, at low fluence the nickel contribution is mediated by a synergistic interaction with the copper precipitate contribution (note the nickel variations were all for high copper steels). Whatever the mechanism, the reduced contributions of copper and nickel are reflected in an inverse temperature dependence of hardness changes below 163°C.

The apparent differences between the temperature dependence of $\Delta\sigma_y$ versus ΔDPH and ΔUTS and the temperature variation in $\Delta\sigma_y / \Delta UTS$ also signal a transition in the mechanical stability of the irradiation-induced microstructures at the low temperature. Specifically, the data suggest that the contribution of small, easily shearable matrix defects may be larger at low temperatures. Such defects could give rise to larger $\Delta\sigma_y$ (a low strain property) without concomitant increases in ΔUTS and ΔDPH (which are high strain properties, as noted previously). This hypothesis is consistent with the expectation that such "small" matrix defects features would exist in higher numbers at the lower temperatures. However, sorting this out will require further detailed microstructural studies, and we have performed some preliminary small angle neutron scattering studies on several materials.

Small angle neutron scattering (SANS) has proven to be a very effective technique for characterizing the ultrafine ($\approx 1\text{nm}$) irradiation induced features which are responsible for hardening and the concomitant irradiation embrittlement of reactor pressure vessel steels. Of course, as is usually the case, full and quantitative characterization of complex (structurally and compositionally) microstructures requires the complementary application of techniques, such as atom probe (AP) imaging and

spectroscopy and high resolution imaging and analytical transmission electron microscopy (TEM/AEM).

Previous studies have clearly demonstrated that at temperatures typical of pressure vessel irradiations ($\approx 300^\circ\text{C}$) copper rich precipitates (CRPs) are a predominant feature in steels bearing significant quantities (>0.05 to 0.1%) of copper. The small (≈ 1 nm) coherent CRPs nucleate quickly and at high number densities from highly supersaturated solutions and grow as a consequence of irradiation-enhanced diffusion. It is also well established that there are other secondary features which play a role in embrittlement. (carbonitrides and phosphides, vacancy clusters dislocation loops). However, the precise nature of these secondary features has yet to be established.

We have carried out a SANS characterization of the irradiation induced fine scale features in alloys irradiated at the lowest (121°C) and highest (288°C) temperatures. The alloy chosen was a 0.4 Cu and 0.8 Ni A533B model steel (LC in Table 3.1.1). The SANS measurements were made by UCSB personnel at the National Institute of Standards and Technology (NIST) on the 8 m instrument. The samples were saturated in a strong (≈ 1.9 T) horizontal magnetic field to separate magnetic and nuclear scattering contributions.

Figure 3.1.7a shows the neutron intensity as a function of the square of the scattering vector for various angles relative to the magnetic field direction for the 288°C irradiation. The data have been normalized to the magnetic scattering intensity using the fit shown in Figure 3.1.7b. The magnetic to nuclear scattering ratio of $M = 1.3$ is consistent with CRPs composed of about $75 \pm 10\%$ Cu alloyed with Mn (and possibly vacancies and Ni). The radius, number density and volume fraction of the CRPs are estimated to be 0.72 ± 0.15 nm, $4.3 \pm 1.9 \times 10^{17}/\text{cm}^3$ and 0.00067 ± 0.00010 respectively. These values are broadly consistent with previous data on the CRP microstructures formed in the early stages of irradiation at around 300°C . In contrast, however, the

Figure 3.1.7 SANS results showing: a) neutron intensity as a function of the square of the scattering vector for various scattering angles and b) fit to the magnetic scattering intensity for alloy LC irradiated at 288°C.

irradiation at 121°C produced no discernable SANS signal, thus supporting the hypothesis, based on the mechanical property data, of a cut-off in precipitation at the lowest temperature.

The failure to detect the features responsible for the small amount of hardening for the 121°C irradiations ($\approx 12 \text{ kg/mm}^2$) can be attributed to the low signal-to-noise ratio. Alternately this may suggest that the hardening is due to small dislocation loops (producing only very weak scattering). The higher hardening in the 288°C irradiation is consistent with the CRP parameters. More recently we have performed microhardness tests on specimens irradiated at 65°C, and the results are consistent with the 121°C data.

We have also performed a post-irradiation annealing study on alloy LC (0.4%Cu-.8%Ni) irradiated at each of the 5 temperatures. Microhardness measurements were made after annealing at 343°C for times ranging from 0.25 to 50 h. Figure 3.1.8 summarizes the results, showing residual irradiation hardness increases as a function of irradiation temperature for the as-irradiated and post-irradiation annealed (0.25h and 35h) conditions. For irradiation temperatures below 163°C and for annealing times greater than about 20h, complete recovery was obtained (0 residual hardness). Recovery of damage at such a low annealing temperature is consistent with the anticipated kinetics for dissolution of vacancy rich clusters or unstable nanovoids. The complete recovery below irradiation temperatures of 163°C suggests that these defects predominate the hardening at these temperatures. The absence of complete recovery at the higher irradiation temperatures is consistent with the presence and hardening contribution of a more stable defect, i.e. CRP's. Again, these results are consistent with the SANS data and the post-irradiation hardening trends described above.

Figure 3.1.8 Residual irradiation-induced hardness change as a function of irradiation temperature for the as-irradiated and post-irradiation annealed (0.25h and 35h at 343°C) conditions for a 0.4%Cu-0.8% Ni alloy.

These preliminary results demonstrate the importance of combining microstructural characterization with mechanical property measurements to developing a physically based understanding of embrittlement. Hence, the experiments we have pursued subsequently and described below should throw considerable additional light on this subject.

3.2 Preparation of Low Flux/Temperature Experiment

3.2.1 Materials

As noted previously, this experiment consists of irradiating mechanical property and microstructural analysis specimens from commercial and model alloys with systematic variations in compositional and metallurgical variables. The materials have been obtained in four sets. The first set consists of 18 simple iron-based model alloys with variations in Cu, Mn, C, Ti and N content. The compositions and alloy codes are given in Table 3.2.1. There are nine alloys in a low nitrogen series and nine in a high nitrogen series, with some Ti additions in the high nitrogen series to examine the gettering effect of Ti on N. Within each series the separate and combined effects of Cu, Mn, and C will be examined. Two additional Fe-high N alloys and two Fe-Ti-low N alloys (one containing copper) were also prepared, but have not been incorporated extensively in the irradiation matrix.

The second set of materials consists of 31 model alloys with an A533B-type base composition, but with significant systematic variations in Cu, Ni, P and Mn content and smaller systematic variations in C, B, N, Al, Mo, Cr, and Group IV elements. Compositions of these alloys are given in Table 3.2.2. The simple model alloys and the A533B-type model alloys were fabricated by Sheffield Labs, UK, in collaboration with AEA Technologies per our specifications. The alloys were made using electrolytic iron and pure alloying elements. Heats were cast into 75mm chill

TABLE 3.2.1
MODEL ALLOY COMPOSITIONS

DESIGNATION	COMPOSITION (weight per cent)				
	Cu	Mn	C	Ti	N(ppm)
VA	--	--	--	--	5
VB	--	1.0	--	--	40
VC	--	1.0	0.15	--	30
VD	0.9	1.0	--	--	10
VE	0.9	1.0	0.15	--	20
VG	0.5	--	--	--	20
VH	0.9	--	--	--	20
VI	0.5	--	0.15	--	10
VJ	0.9	--	0.15	--	10
VK	--	--	--	--	100
VL	--	--	--	0.25	100
VM	0.5	--	--	--	120
VN	0.9	--	--	--	110
VO	0.9	--	0.15	--	110
VP	0.9	--	--	0.25	110
VR	--	1.0	--	--	110
VS	0.9	1.0	--	--	110
VT	0.9	1.0	--	0.25	90
VW	--	--	--	--	80
VV	--	--	--	--	120
(VX)	--	--	--	0.25	20
(VY)	0.9	--	--	0.25	20

TABLE 3.2.2
COMMERCIAL MODEL ALLOYS

COMPOSITION (weight per cent)												
ALLOY	Cu	Ni	Mn	Cr	Mo	P	N*	As/Sb Sn each	B*	C	Al	Si
C-1	0	0	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-2	0	0	1.4	0	0.5	0.04	<50	<0.005	<1	0.15	.005	0.25
C-3	0	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-4	0	0.8	1.4	0	0.5	.025	<50	<0.005	<1	0.15	.005	0.25
C-5	0	0.8	1.4	0	0.5	0.04	<50	<0.005	<1	0.15	.005	0.25
C-6	0	1.6	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-7	0	1.6	1.4	0	0.5	0.04	<50	<0.005	<1	0.15	.005	0.25
C-8	0	0.8	0	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-9	0	0.8	0.7	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-10	0.05	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-11	0.3	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-12	0.8	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-13	0.1	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-14	0.1	0.8	1.4	0	0.5	0.04	<50	<0.005	<1	0.15	.005	0.25
C-15	0.2	0	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-16	0.2	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-17	0.2	1.6	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-18	0.4	0	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-19	0.4	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-20	0.4	1.6	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-21	0.4	0.8	0	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-22	0.4	0.8	0.7	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-23	0	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.05	.005	0.25
C-24	0	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.30	.005	0.25
C-25	0	0.8	1.4	0	0.5	.005	200	<0.005	<1	0.15	.005	0.25
C-26	0	0.8	1.4	0	0.5	.005	<50	0.02	<1	0.15	.005	0.25
C-27	0	0.8	1.4	0	0.5	.005	<50	<0.005	10	0.15	.005	0.25
C-28	0.4	0.8	1.4	0	0.5	.005	<50	<0.005	10	0.15	.005	0.25
C-29	0.2	0	1.4	0	0	.005	<50	<0.005	<1	0.15	.005	0.25
C-30	0.2	0.8	1.4	0.4	0.5	.005	<50	<0.005	<1	0.15	.005	0.25
C-31	0	0.8	1.4	0	0.5	.005	<50	<0.005	<1	0.15	.005	0.25

* in atom parts per million

molds, then forged at 1100°C to approximate dimensions, finish forged at 850°C, and air cooled.

The third set consists of another set of A533B-type steels that were used in several previous irradiation programs[5]; eight quench-and-tempered, bainitic alloys were selected that have the following variations in Cu and Ni content:

ALLOY	COMPOSITION (%)		RATIONALE
	Cu	Ni	
LA	.4	.001	Nickel sequence
LB	.4	.175	"
LJ	.4	.8	"
LD	.4	1.25	"
LG	.005	.8	Copper sequence
LH	.110	.8	"
LI	.20	.8	"
LK	.8	.8	"

These provide some connection to data sets obtained with these alloys at higher irradiation temperatures.

The fourth set of materials consists of eight commercial alloys: an A588B steel supplied by NII of the UK; an A36 steel; both HFIR A212 and A350 steels obtained from both archival material as well as irradiated surveillance specimens and supplied by ORNL; and the Shippingport reactor neutron shield tank material obtained from both the inner wall (irradiated) and outer wall (effectively unirradiated).

For purposes of specimen fabrication and heat treatment (except for the irradiated materials), we sectioned coupons from the material by electro-discharge machining (EDM) with dimensions described below. Coupons were clamped in a steel jig during heat treatment to maintain flatness and to control heating and cooling rates. The simple model alloys listed in Table 3.2.1 were given a solution treatment at 775°C for 17h, followed by quenching in a salt bath at 450°C for 3 minutes and a subsequent air cool. This heat treatment ensures that the copper additions are all initially in solution

(no pre-precipitation), that the microstructure is medium to large grained ferrite, and that no unstable carbides or nitrides are produced upon quenching, obviating the need for any additional heat treatment. The absence of an unstable microstructure and the presence of copper in solution was confirmed by a series of post-heat treatment aging studies performed by AEA Technologies. Alloys were subjected to periodic agings at 550°C for times up to 1000 hours. With the exception of the Ti-bearing alloys, the non-copper bearing alloys showed no significant gains or losses in hardness, indicative of a stable microstructure. The 0.9 Cu alloys showed a characteristic peak in hardness at about 5h, as shown in Figure 3.2.1a, indicative of precipitation of Cu initially in solution. Interestingly, the Ti-bearing alloys (both with and without copper) showed evidence of hardening beyond 100h, as shown in Figure 3.2.1b. This is presumably due to precipitation of TiN, and this will be verified by microstructural analysis. However, in the C-bearing alloys, there is an absence of a discernible yield point and Luders plateau. This suggests that the cooling rates were too fast for C to segregate to dislocation lines. Indeed, an age of 550°C for 24 h established a yield point and Luders plateau -- i.e., produced strain aging. While this has little impact on the low temperature irradiations pursued here, it could necessitate an additional strain age for any future high temperature irradiations of these alloys.

The A533B-type alloys were given a number of different heat treatments. All of the alloys were given the following base heat treatment: austenitize at 950°C for 0.5h; quench in a salt bath at 450°C for 10 minutes followed by an air cool; temper at 660°C for 4 h followed by an air cool. These conditions -- determined by an iterative combination of analysis and experiment -- were chosen to initially solutionize the alloying elements and to provide for a fully tempered bainitic structure without pre-precipitating the copper in solution. However, since some of the compositional variations (e.g., Cu, Ni, Mn and C) can also significantly affect the time-temperature-

Figure 3.2.1 Hardness as a function of aging time at 550°C for a) an 0.9Cu model alloy and b) a high N, Ti-bearing model alloy

transformation envelope (hence the degree and type of bainite transformation), we added an additional set of heat treatments for selected alloys to cross reference initial properties and microstructures against this effect; hence for alloys C-19 (0.4 Cu, 0.8 Ni) and C-20 (0.4 Cu, 1.6Ni) we performed an additional heat treatment in which the quench bath temperature was increased to 500°C; and for alloys C-8 (0 Cu, 0.8 Ni, 0 Mn) and C-23 (0 Cu, 0.8 Ni, 0.05 C) we performed an additional heat treatment in which the quench bath temperature was decreased to 400°C.

To examine the effect of pre-precipitation of copper on the irradiation hardening response, we added a set of aging heat treatments to several copper-bearing alloys to promote different extents of pre-precipitation. These are summarized below:

ALLOY (Cu/Ni)	HEAT TREATMENT
C-19 (.4Cu/.8Ni)	Base + 8h @ 600°C Base + 40h @ 600°C Base + 80h @ 600°C
C-18 (.4Cu/0Ni)	Base + 40h @ 600°C
C-20 (.4Cu/1.6Ni)	Base + 40h @ 600°C
C-12 (.8Cu/.8Ni)	Base + 8h @ 600°C

To examine the effects of prior austenite grain size on the hardening response, we performed an additional heat treatment on alloys C-15 (0.2 Cu, 0Ni), C-16 (0.2 Cu, 0.8 Ni), C-17 (0.2 Cu, 1.6 Ni), and C-19 (0.4 Cu, 0.8 Ni) where the austenitization conditions were increased to 1200°C for 1h versus the base condition of 950°C for 0.5h and the quench and temper conditions were held constant. Finally, to examine the effects of impurity segregation at grain boundaries we aged an additional set of base-heat treated alloys -- C-5 (0Cu, 0.8Ni, 0.04P) and C-26 (0 Cu, 0.8 Ni, 0.02 Group IV elements) -- at 480°C for 100h.

The commercial alloys were used in the as-received condition except for the Shippingport inner wall, the HFIR surveillance materials (A212 and A350), and several alloys we had in our archives from a previous irradiation program (LA, LC, LD). For the irradiated materials, 0.5mm thick 10mm x 10mm coupons were mechanically wafered from available materials, and these were post-irradiation annealed according to the following schedule to recover the irradiation damage:

ORNL A 350:	annealed at 450°C for 50 h
	annealed at 600°C for 5 h
	annealed at 343°C for 290 h
ORNL A 212:	annealed at 450°C for 50 h
	annealed at 600°C for 5 h
	annealed at 343°C for 290 h
SHIPPINGPORT:	annealed at 450°C for 50 h
	annealed at 600°C for 5 h
	annealed at 343°C for 290 h
LAVAL LA70:	annealed 600°C for 5 h
LAVAL LC70:	annealed 600°C for 5 h
LAVAL LD70:	annealed 600°C for 5 h
LAVAL LG70:	annealed 600°C for 5 h

Microhardness tests were performed on the coupons before and after annealing to compare to the subsequently irradiated material.

For all of the unirradiated materials, 0.635mm thick EDM'd coupons were lapped to a thickness of $0.51 \pm .02$ mm. From these lapped coupons we punched miniature tensile specimens with the geometry shown in Figure 3.2.2 and 3mm diameter discs. The disc specimens can be used for both microhardness and transmission electron microscopy. Enough specimens of each type were punched for a

A = 9.0 mm	L = 27.51 mm	Tolerance on all dimensions = ± 0.025 mm
B = 7.75 mm	T = 0.51 mm	
C = 5.0 mm	W = 2.0 mm	
D = 1.50 mm	R = 1.50 mm	
E = 3.89 mm		

Surface Condition:

#32 microfinish all surfaces

Labels:

X - Two digit alloy I.D. #
 Y - Two digit condition I.D. #
 Z - One digit specimen #

Labels are laser engraved.

Figure 3.2.2 Schematic illustration of miniature tensile specimen geometry.

redundancy of 3 in each of the irradiation experiments. The punch and die used to punch the miniature tensile specimens was checked for wear by punching a set of standard specimens (mild and stainless steel) periodically and testing them; signs of wear were indicated first by decreases in the ductility of the stainless steel standards, well before any changes in strength (yield or ultimate) of either material, and hence the punch and die were resharpened at the first signs of wear.

In addition to these specimens, we also fabricated small angle neutron scattering (SANS) specimens from selected alloys. The SANS matrix for this experiment is comprised of two parts. The first part consists of the following alloys (numbers correspond to Table 3.2.2): C-1, C-3, C-4, C-5, C-6, C-12, C-13, C-16, C-18, C-19, C-20, C-21, and C-22. These were chosen on the basis of the following rationale:

- Alloys C-3, C-13, C-16, and C-19 comprise a sequence of copper variations.
- Alloys C-18, C-19, and C-20 comprise a sequence of nickel variations at high copper.
- Alloys C-1, C-3, and C-6 comprise a sequence of nickel variations at low copper
- Alloys C-19, C-21, and C-22 comprise a sequence of manganese variations.
- Alloys C-3, C-4, and C-5 comprise a sequence of phosphorous variations.
- Alloys C-18, C-19, C-20, and C-12 will be used to examine the effect of pre-irradiation aging (copper in solution).

Cross comparisons will also permit analysis of some synergistic effects: for example, Cu-P and Ni-P-Cu. These alloys were all given the base heat treatment, and again selected alloys were given an additional heat treatment to examine the effect of copper pre-precipitation:

ALLOY (Cu/Ni)	HEAT TREATMENT
C-19 (.4Cu/.8Ni)	Base + 8h @ 600°C Base + 40h @ 600°C Base + 80h @ 600°C
C-18 (.4Cu/0Ni)	Base + 40h @ 600°C
C-20 (.4Cu/1.6Ni)	Base + 40h @ 600°C
C-12 (.8Cu/.8Ni)	Base + 8h @ 600°C

Some FIM wires were also fabricated from these alloys on an as-available basis. The second part of the SANS matrix consists of selected simple model alloys and commercial archival material. The model alloys are VA, VB, VC, VD, VF, VG and VK; these were selected on the basis of the following rationale:

- Alloys VA, VG, and VH comprise a sequence of copper variations.
- Alloys VD and VH comprise a sequence of manganese variations at fixed copper.
- Alloys VA and VJ comprise a sequence of nitrogen variations.
- Alloys VB and VC comprise a sequence of carbon variations at fixed manganese.

These have been heat treated in the same manner as the tensile coupons of the simple model alloys. The commercial archival specimens consist of the HFIR A212 and Shippingport shield tank outer wall. The SANS specimens consist of lapped 19 x 20 mm rectangular coupons with thicknesses ranging from 0.5mm to 1.3mm.

In summary, the metallurgical/specimen matrix assembled for irradiation comprises the largest experimental matrix with systematic variations in composition and metallurgical conditions for the purpose of investigating radiation hardening/embrittlement that we are aware of.

3.2.2 Irradiation Capsules and Conditions

The irradiation capsule consists of an inner and outer box. The outer box is a hollow rectangular aluminum box with 3.175 mm thick walls and an internal cavity dimension of 83 x 48.2 x 17.7 mm with top and bottom aluminum caps. The purpose of this box is to provide a hermetically sealed container for the inner box to separate the specimens from the test reactor water coolant. The inner box consists of an internal frame to hold various specimen geometries and dosimetry foils (described later) and an upper and lower lid. The geometry of the internal frame is shown in Figure 3.2.3. All parts are comprised of 1100 aluminum. Following loading, the specimen and dosimetry foil stacks are overlaid with shim stock to achieve an exact height and hence hard contact with the upper and lower lid (for heat transfer purposes). The top lid is attached by screws, and the whole assembly is machined to achieve a tight fit in the outer box cavity. Prior to loading the outer box, the lower cap is welded on. Following loading, the outer box is evacuated and back filled with He, and the top lid is welded on. The entire assembly is then He leak-checked to ensure hermeticity.

The irradiation matrix is shown in Table 3.2.3 along with the reactor facility. As shown in Figure 3.2.4, the Ford Reactor irradiations are performed at peripheral locations. An external grid plate has been constructed to hold sample holder tubes which resemble fuel-tube geometry, and we have modified 3 tubes for purposes of irradiating our capsules. The geometry of these tubes is shown in Figure 3.2.5. The inner diameter of each tube has been lined with a 0.030 inch thick layer of Cd to reduce the thermal neutron flux, largely to minimize radioactivation of the specimens for post-irradiation testing purposes. The Cd layer is overlaid with an inner wrapper of aluminum sheet. We have fabricated a capsule carrier that slides into the inner diameter of this lined tube. The capsule carrier features an internal track that provides a means of holding the capsule train at an elevation corresponding to the desired flux level.

Irradiation Capsule For The Univ. of Michigan Reactor

Figure 3.2.3 Schematic illustration of the inner capsule geometry.

Table 3.2.3
Target 60°C Irradiation Conditions

Flux (n/cm ² -s)	Fluence (n/cm ²)				Reactor
	2 x 10 ¹⁷	5 x 10 ¹⁷	3 x 10 ¹⁸	7 x 10 ¹⁸	
2 x 10 ¹⁰	F1 ^a	F4 ^a	---	---	Ford
2 x 10 ¹¹	F2 ^b	F5 ^b	F7 ^a	---	Ford
7 x 10 ¹¹	--	F6 ^b	F8 ^b	F9 ^b	Ford
3 x 10 ¹³	--	--	F10 ^b	---	HFBR
5 x 10 ¹⁴	---	F11 ^b	F12 ^b	---	HFIR
5 x 10 ¹¹	---	F13 ^{b,c}	---	---	HFBR

a -- full capsule
 b -- partial capsule
 c -- thermal/fast ~370

Figure 3.2.4 Schematic illustration of the Ford Reactor irradiation geometry.

Figure 3.2.5. Schematic illustration of capsule holder tubes.

Irradiations to various fluences at a single flux level are achieved by inserting all of the capsules initially, and removing them sequentially as each target fluence is achieved. Capsule dimensions were sized in order to achieve uniform irradiation of the entire capsule chain within $\pm 10\%$ variation. A stepped recess was machined on the exterior of each capsule. A retraction tool we have designed and built can be inserted down the sample carrier tube; this locks onto the recesses to allow removal of individual capsules when they reach their desired fluence. We have run instrumented mock-up capsules in the Ford reactor to determine the extent of gamma heating. The data from these experiments indicate that temperature variations are less than 2°C over the entire capsule thickness even at the highest flux position. Hence, for all intents and purposes, the irradiations will be conducted at water temperature. As an insurance measure, we also added melt wires to several capsules.

Dosimetry is achieved in a collaborative program with ORNL. Dosimetry packages in each capsule include the following materials: U-238, Ni, Fe, Ti, Cu, Co-Al, Nb, and Al_2O_3 . Following irradiation, the dosimetry packages are analyzed by ORNL in exchange for providing piggyback irradiation space in our tubes.

3.2.3 Post Irradiation Testing

As indicated above, we rely on both microhardness and miniature tensile tests to track mechanical property changes in these materials, and we have improved our post-irradiation testing capabilities for both of these techniques in anticipation of the large number of specimens that will be generated in this experiment.

We have developed several generations of automated Vickers microhardness testers. The most recent version is based on a LECO M400A microhardness tester, which has a robust, direct load train for applying load to the indenter. This is mounted on a vibration isolation table, which damps vibration amplitudes by 10-100 times in the

10-100 Hz range. An X-Y specimen stage is driven by two stepper motors with 5 μm resolution. The motion of the stage and the indentation sequence is computer-controlled (PC-AT-compatible), so any number and spatial sequence of indentations can be programmed for automated indentation. We use a 500g load at room temperature and run an indentation sequence overnight for next day's analysis. Subsequently, the indentations are examined under a microscope in the same sequence; the microscope image is transferred by videocamera to a monitor, and the image is digitized by a framegrabber in the same computer that controls the indentation process. An option also exists to enhance the contrast of the digitized image. The user can select a number of analysis techniques that we have developed. The one we have found to be most reliable is a user-interactive technique whereby the user moves a mouse-controlled cursor on the monitor screen to a corner of the indentation, magnifies the pixel array in the vicinity of this corner (by clicking the mouse), and selects the magnified pixel corresponding to the corner (again by clicking the mouse); after repeating this at the other three corners, the software automatically calculates the Vickers microhardness number and stores this in a data base.

We have also developed a number of different strategies for holding specimens. The bulk of our testing is performed on 3mm diameter discs 0.5mm in thickness of the type prepared for this experiment. For pressure vessel steels, indentations are typically 60 μm across, and hence we can perform up to 61 indentations on a single specimen without violating rules of thumb for eliminating proximity effects from neighboring indentations or specimen edges. Such a large number of indentations is attractive, for instance, for performing post-irradiation annealing experiments. Grouping specimens is desirable for automated testing, and while specimens can be mounted in groups in a standard epoxy or phenolic metallurgical mount, we have developed an additional

holder to permit ease of removal after testing as well as elevated temperature annealing without removal from the holder, which is not possible with polymeric metallurgical mounts. Such a capability allows us to perform several anneals (e.g., an isothermal sequence) on the same set of specimens. The holder is a rectangular block (both aluminum and stainless steel are used) machined with recesses to hold the discs in the desired pattern; the block is also slotted to produce tines with through holes to permit clamping the specimens in place by tightening screws in the through holes and squeezing the tines together. Since it is desirable to maintain flatness across the entire group of specimens for subsequent indentation and analysis, the entire assembly is ground and polished through 0.5 μm Al_2O_3 after it is loaded.

We have recently automated our tensile testing apparatus to permit rapid testing of a large number of irradiated tensile specimens. A schematic illustration of some of the components of this scheme is shown in Figure 3.2.6. The apparatus is based on an Instron 1122 screw-driven machine, which we have modified to allow computer control. A cassette of up to 25 specimens is loaded (in a remote warm cell, if they are irradiated), and bolted to a linear table at the base of the Instron. Again by computer control, the table is driven to position the first specimen between the faces of a hydraulically loaded upper grip. Upon command from the operator, the grip is closed, the cross head activated and the specimen pulled to failure. Load-displacement data are logged by the computer throughout the duration of the test. Following failure, the upper grip faces are released, the cross head lowered to its initial position, and the next specimen positioned for subsequent testing. We have also written software to permit analysis of the load-displacement data to produce stress-strain curves; to determine values of yield stress; ultimate tensile strength, uniform and total ductility; and to load these values into a data base. We have fit the stage with a protective box to permit testing radioactive specimens.

Figure 3.2.6. Schematic illustration of the automated tensile test system.

3.3 Summary of Results

A subset of the alloys have been irradiated in the University of Michigan Ford Reactor at a flux of $2 \times 10^{11} \text{n/cm}^2\text{-s}$ to fluences of 0.2 and $0.5 \times 10^{18} \text{n/cm}^2$. The capsules contained minitensiles of 12 alloys in the base heat treated condition: CM1, 3, 5, 6, 11, 12, 13, 16, 18, 19, 20 and 22. These alloys had a range of copper contents (0 - 0.8%), nickel contents (0 - 1.6%) and high and low manganese and phosphorous contents. Post-irradiation tensile testing has been performed on one specimen of each of the materials.

These data are plotted in Figures 3.3.1 to 3.3.3. Figure 3.3.1 shows the variation of yield stress changes with copper and nickel for the $0.5 \times 10^{18} \text{n/cm}^2$ irradiation. These data suggest a relatively weak dependence of hardening on nickel, manganese and phosphorous at this low fluence, and perhaps even an "inverse" dependence on copper. Figure 3.3.2 shows the variation of yield stress change with the square root of fluence; Figure 3.3.2 a) shows this variation for alloys with various copper contents at intermediate nickel (0.8%); and Figure 3.3.2b) shows this variation for alloys with various nickel contents at low copper (nominally 0%). The data corresponding to the high phosphorous alloy are also indicated. Again the dependence on nickel and phosphorous appear weak, and a possible inverse dependence on copper is suggested but only at the higher fluence. In all cases a square root of fluence dependence of hardening is apparent. Figure 3.3.3 shows these data in comparison to other low temperature irradiation hardening data. Here yield stress changes are plotted against the square root of fluence. The open circles represent averages of the data obtained in this program on the 12 alloys just described. The open squares and triangles represent averages of the data described in section 3.1. The solid circles represent data obtained in an ORNL irradiation in HFIR at ambient temperature and a

Figure 3.3.1 Variation of yield stress changes with copper and nickel for the $0.5 \times 10^{18} \text{ n/cm}^2$ irradiation.

Figure 3.3.2 Variation of yield stress change with the square root of fluence: a) for alloys with various copper contents at intermediate nickel (0.8%); and b) for alloys with various nickel contents at low copper (nominally 0%).

Figure 3.3.3 Yield stress changes versus the square root of fluence. The open circles represent averages of the data obtained in this program on the commercial model alloys. The open squares and triangles represent averages of the data described in section 3.1. The solid circles represent data obtained in an ORNL irradiation in HFIR at ambient temperature and a higher flux ($450 \times 10^{12} \text{ n/cm}^2\text{-s}$). Figure 3.3.3(b) is a magnification of the low fluence portion of Figure 3.3.3a).

Figure 3.3.4 Variation of yield stress change with square root of fluence for a) A212, b) A350 and c) A533B steels irradiated in HFIR (450×10^{12} n/cm²-s) ORR (12.6×10^{12} n/cm²-s) and lower flux (MTR, surveillance) irradiations at ambient temperatures.

Figure 3.3.5 Fluence (in units of 10^{18} n/cm²) required to achieve a hardening of 50 MPa versus the log of the flux (in units of 10^{12} n/cm²-s).

higher flux ($450 \times 10^{12} \text{ n/cm}^2\text{-s}$). Figure 3.3.3b) is a magnification of the low fluence portion of Figure 3.3.3a). Taken as a whole, these data also suggest a dependence of hardening on the square root of fluence at these low temperatures, and a relatively weak dependence on flux in this temperature, flux and fluence regime.

For comparison, data on A212, A350 and A533B steels irradiated in HFIR ($450 \times 10^{12} \text{ n/cm}^2\text{-s}$), ORR ($12.6 \times 10^{12} \text{ n/cm}^2\text{-s}$) and lower flux (MTR, surveillance) irradiations at ambient temperatures are shown in Figure 3.3.4.[10,14,15] The average data for 0.1 and 0.2 Cu steels obtained in this program are superimposed on the A533B data, and the average tensile data obtained on the A212 and A350 steels, as described in section 3.1, are shown on the corresponding plots. Again the hardening dependence on square root of fluence is apparent as well as the relative insensitivity to flux down to levels of about $0.2 \times 10^{12} \text{ n/cm}^2\text{-s}$. Below this, the relatively few data points available suggest that lower flux may lead to greater hardening. This is shown to a greater extent in Figure 3.3.5 where these data are cross plotted to show the fluence required to achieve a hardening of 50 MPa versus the log of the flux. These data suggest that above about $10^{11} \text{ n/cm}^2\text{-s}$ the dependence of hardening on flux is small, but at lower fluxes the hardening may be accelerated somewhat. A key to addressing this question will be the set of irradiations in the University of Michigan reactor at the low flux range ($\sim 2 \times 10^{10} \text{ n/cm}^2\text{-s}$)

Hence, it is of significant importance to continue this irradiation program under a follow-on activity. In addition to the two irradiations described above, one full set of specimens has already been irradiated at $7 \times 10^{11} \text{ n/cm}^2\text{-s}$ to a fluence of $3 \times 10^{18} \text{ n/cm}^2$. These are currently cooling. Three more full sets of specimens have been left in reactor to receive irradiation at a flux level of $\sim 0.02 \times 10^{12} \text{ n/cm}^2\text{-s}$ to fluences of 2 and $5 \times 10^{17} \text{ n/cm}^2$ and at a flux level of $2 \times 10^{11} \text{ n/cm}^2\text{-s}$ to a fluence of $3 \times$

10^{18}n/cm^2 . A follow-on effort should be funded to complete these irradiations. In addition, analysis of the data from this program support examining hardening at the following remaining combinations of flux and fluence:

<i>Flux</i> ($10^{12}\text{n/cm}^2\text{-s}$)	<i>Fluence</i> (10^{18}n/cm^2)	<i>Reactor</i>
0.7	7	Ford
0.7	0.5	Ford
30	3	HFBR
500	0.5	HFIR
500	3	HFIR
0.5*	0.5	HFBR

The last irradiation (*'d) should be performed at a high ratio of thermal to fast neutrons to address the question of thermal neutron contributions to hardening. Combined with the data generated in this program, these irradiations should provide a definitive data base for addressing low flux, low temperature hardening and embrittlement.

REFERENCES

1. R. K. Nanstad, S. Iksander, A. Rowcliffe, W. Corwin, G. R. Odette, *ASTM-STP-1046*, (1990) 5-29.
2. A. F. Rowcliffe, G. R. Odette, et al., Section 6, Radiation Effects and Mechanistic Studies, in: "Evaluation of HFIR Pressure Vessel Integrity Considering Radiation Embrittlement," *ORNL/TM-10444* (1987).
3. R. D. Cheverton, W.E. Pennell, G. C. Robinson, R. K. Nanstad, *NUREG/CR-5320*, Nuclear Regulatory Commission, January 1989.
4. R. H. Priest, W. Charnock, and B. K. Neale, "Effects of Irradiation on Materials," *ASTM-STP-870* (1985) 1150.
5. G. R. Odette and G. E. Lucas, "Irradiation Embrittlement of LWR Pressure Vessel Steels, *EPRI NP-6114*, Electric Power Research Institute, January 1989.
6. G. E. Lucas, G. R. Odette, P. M. Lombrozo, J. W. Sheckherd, *ASTM-STP-870* (1985) 900.
7. G. R. Odette and G. E. Lucas, 3rd Intl. Symp. Env. Dg. Materials (1987).
8. G. R. Odette and G. E. Lucas, *ASTM-STP-909* (1986) 206.
9. G. E. Lucas, G. R. Odette, R. Maiti, J. W. Sheckherd, *ASTM-STP-956* (1988) 379.
10. J. R. Hawthorne, S.T. Rosinski, "Accelerated 54°C Irradiated Test of Shippingport Neutron Shield Tank and HFIR Vesel Materials," *SAND92-2420*, Sandia National Labs, 1993.
11. J. Stiegler, J. Weir, Metals and Ceramics Division Progress Report, *ORNL-6705*, Oak Ridge National Lab, 1992.
12. G. R. Odette, P. M. Lombrozo, R. A. Wullaert, *ASTM-STP-870* (1985) 840.
13. Minutes of the Plant Lifetime Extension Improvement Meeting, Sandia National Lab, 9/17-18/87.
14. K. Farrell, S. T. Mahmood, R. E. Stoller, L.K. Mansur, NRC Water Reactor Safety Meeting, 1992.
15. R. D. Cheverton, J.G. Merkle, R.K. Nanstad, "Evaluation of HFIR Pressure-Vessel Integrity Considering Radiation Embrittlement," *ORNL/TM-10444*, Oak Ridge National Lab, 1988.

**DATE
FILMED**

7/14/94

END