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Neutron Irradiation Embrittlement of Several Higher Strength Steels

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Metallurgy Division

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Abstract: Several steels representative of recently developed types and having potential for nuclear structural applications were exposed to high energy nuclear radiation, and the resultant properties were compared with those of the currently used A212-B and A302-B nuclear reactor pressure vessel steels. Preliminary results from several comparative irradiation experiments indicate that certain higher strength steels, in addition to having initial qualities of higher strength and lower initial ductile-brittle transition temperatures, show smaller embrittlement, earlier embrittlement saturation, and a superior overall response to irradiation at 550°F than that observed for the steels in current reactor pressure vessels.

INTRODUCTION

Higher strength steels are being considered for future reactor pressure vessel construction, because the ever-growing size of nuclear reactors is placing severe demands on the widely used A302-B pressure vessel steel—requiring its use in thicknesses of 12 inches or greater for some advanced pressurized-water reactors. Along with higher strength, steels are desired which exhibit lower sensitivity to neutron embrittlement. With these two factors in mind, certain higher strength steels were added to the NRL irradiation effects studies.

Following the procedures of earlier neutron embrittlement studies, the increase in the ductile-brittle transition temperature or in the nil-ductility transition (NDT) temperature as determined by Charpy V-notch tests formed the basis for comparing the higher strength steels with the ASTM reference steels, A212-B and A302-B. For a screening comparison of several steels after simultaneous irradiation, the preliminary experiments used sealed capsule assemblies of relatively simple design permitting irradiation of many specimens at reactor ambient temperatures. Later experiments involved the more complex irradiation units in which the exposure temperature was controlled at 550°F to more closely simulate the operating condition of current reactor pressure vessels.

The higher strength steels selected for study exhibited yield strength levels from about 80 to 180 ksi and were of different alloying chemistry ranging from the relatively lean composition of the SSS-100 to the heavily alloyed maraging steels.

This preliminary program which emphasized comparative evaluations of neutron embrittlement among steels of several strength levels also permitted the assessment of the effects of progressively higher neutron exposure, the benefits of irradiation at 550°F *versus* lower temperature levels, and the possible benefits of low initial NDT values of these steels in terms of the maximum embrittlement observed.

The results obtained suggest that several of the higher strength steels have points of great merit for nuclear service application when they are compared with the performance of the A212-B and A302-B steels. In general, the present irradiation study indicates great promise for the higher strength steels.

IRRADIATION OF STEELS

Comparative irradiation experiments were conducted in three light-water-moderated test reactors. The Oak Ridge Low Intensity Test Reactor (LITR) and the Union Carbide Research Reactor (UCRR) were used for the low neutron exposure-low temperature irradiations, while the Materials

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NRL Problem M01-14; Projects SF 020-01-05-0858; RR 007-01-46-5409; AT(49-5)-2110; and USA-ERG-4-66. This is an initial report on one phase of the overall problem; work on this and other phases is continuing. Manuscript submitted April 8, 1966.

NOTE: This report will be published by the ASTM as an original presentation at the Third International Symposium on the Effects of Radiation to Structural Materials, Atlantic City, New Jersey, June 1966.

TABLE 1A
Chemical Composition of Steels in the Comparative Irradiation Study

Type of Steel	Chemical Analysis (%)												
	C	Mn	Si	P	S	Ni	Cr	Mo	Al	V	N	Cu	Ti
A212-B	0.26	0.76	0.24	0.011	0.031	0.22	0.20	0.02	—	—	—	—	—
A302-B	0.20	1.31	0.25	0.012	0.023	0.20	0.17	0.47	—	—	—	—	—
3-1/2Ni-Cr-Mo	0.17	0.38	0.29	0.013	0.023	3.65	1.88	0.51	0.02	—	0.010	—	—
7-1/2Ni-Cr-Mo	0.12	0.27	0.21	0.008	0.008	7.38	0.81	0.97	0.034*	—	—	—	—
A353 (Q and T)	0.09	0.44	0.21	0.009	0.014	8.85	0.04	0.02	0.011	—	—	—	—
A387-B	0.12	0.49	0.23	0.010	0.019	0.10	1.02	0.52	0.04	0.003	—	0.19	0.004
A387-D	0.14	0.30	0.24	0.012	0.020	—	2.05	0.98	—	—	—	—	—
SSS-100	0.18	0.51	0.28	0.017	0.020	0.08	1.72	0.50	0.036	—	—	0.26	0.085

*Soluble aluminum.

TABLE 1B
Heat Treatment of Steels in the Comparative Irradiation Study

Type of Steel Plate	Thickness (in.)	Heat Treatment
A212-B	4	Austenitized at 1650°F for 2 hr; water quenched; tempered at 1175°F for 4 hr; furnace cooled to below 600°F.
A302-B	6	Austenitized at 1650°F for 2 hr; water quenched; tempered at 1200°F for 6 hr; furnace cooled to below 600°F.
3-1/2Ni-Cr-Mo	8	Austenitized at 1650°F for 8 hr heating and 2 hr hold; water quenched for 17 min; reaustenitized at 1500°F with 8 hr heating and 2 hr hold; water quenched for 17 min; tempered at 1185°F with 8 hr heating and 2 hr hold; water quenched cold.
7-1/2Ni-Cr-Mo	1	Austenitized at 1550°F for 1 hr; water quenched; tempered at 1025°F for 1 hr; water quenched.
A353 (Q and T)	2	Austenitized at 1475°F for 2 hr; water quenched; tempered at 1100°F for 2 hr; water quenched.
A387-B	6	Normalized at 1640-1680°F for 6-1/2 hr; air cooled; tempered at 1340-1370°F for 6 hr; air cooled; stress relieved at 1150-1175°F for 6 hr; furnace cooled to 600°F.
A387-D	6	Normalized at 1700°F for 6 hr; air cooled; tempered at 1350°F for 6 hr; air cooled.
SSS-100	6	Austenitized at 1650°F; water quenched; tempered at 1225°F for 1-1/2 hr; retempered at 1240°F for 2 hr.

TABLE 1C
Mechanical Properties of Steels in the Comparative Irradiation Study

Type of Steel	Charpy-V 30-ft-lb Transition Temp. (°F)	Yield Strength (0.2% Offset) (ksi)	Tensile Strength (ksi)	Elongation (1-in. gage length) (%)	Reduction of Area (%)
A212-B	5	44.7	79.1	33.6	64.6
A302-B	30	70.7	92.3	27.2	64.8
3-1/2Ni-Cr-Mo	−130	95.0	122.0	24.7	68.7
7-1/2Ni-Cr-Mo	−215	148.8	169.4	19.0	68.7
A353	−370*	103.0	112.6	27.1	69.1
A387-B	30	37.2	61.4	38.7	75.3
A387-D	55	42.4	71.1	33.4	72.0
SSS-100	−45	97.5	110.9	22.3	68.9

*Extrapolated value.

Testing Reactor (MTR) at the National Reactor Test Station was used for the high neutron exposure irradiation. The controlled temperature (550°F) irradiations were conducted in the LITR. The irradiation techniques have been described previously (1).

The A212-B and A302-B steels were used as the reference steels because of the wealth of post-irradiation data and the broad pressure vessel application which these two steels have received. Simultaneous irradiations of the higher strength steels were conducted with either the A212-B or the A302-B steel or both included as controls. The steels studied included 3-1/2Ni-Cr-Mo, A353, SSS-100, and 7-1/2Ni-Cr-Mo steels having yield strength levels ranging from 90 to 150 ksi plus the lower strength (~40 ksi yield strength) A387 Grades B and D steels. In addition to results on these steels, some preliminary data have also been obtained from one experiment conducted in the UCRR involving similar comparative irradiations of the 160 to 180 ksi yield strength 12Ni-5Cr-3Mo maraging steel and a 9Ni-4Co-.25C steel. While obviously not all inclusive, this preliminary series of comparative irradiation experiments does provide an opportunity for comparing the relative radiation embrittlement sensitivity of several promising classes of higher strength steels.

The composition and heat treatment information for the 90 to 150 ksi strength group, as well as the A387 steels and the A212-B and A302-B reference steels, is presented in Tables 1A and 1B; the pertinent initial mechanical properties are given in Table 1C. Similar data for the higher strength (160 to 185 ksi) maraging and nickel-cobalt steels are presented in a later section.

EXPERIMENTAL OBSERVATIONS

In order to introduce as little experimental variation as possible, the basis of comparison for this report involves the simultaneous irradiation of the several steels being compared. However, it was not possible to include every steel in each of the irradiation experiments upon which this limited comparative review is based. The comparative irradiation experiments included in this report covered a range of neutron exposure levels from about 1×10^{19} to 1×10^{20} n/cm^2 (>1 Mev), though most of the exposures were less than 4×10^{19} n/cm^2. All neutron exposures were determined by analysis of induced Mn-54 activity in Fe flux monitor wires, with the assumption of a fission neutron spectrum at the irradiation facility.

As indicated above, a large amount of irradiation embrittlement data has been obtained in the past for A212-B and A302-B steels. The results for these two steels have been presented in detail in earlier reports (2,3). In order to facilitate comparisons and to provide a frame of reference for subsequent discussions, however, the irradiation embrittlement results for one heat of A302-B are summarized in Fig. 1. This original reference heat was produced by the U.S. Steel Corporation and distributed under the auspices of the ASTM (4). The graph may be considered typical of the behavior trends for other heats of A302-B steel as well as for the A212-B reference steel, both of

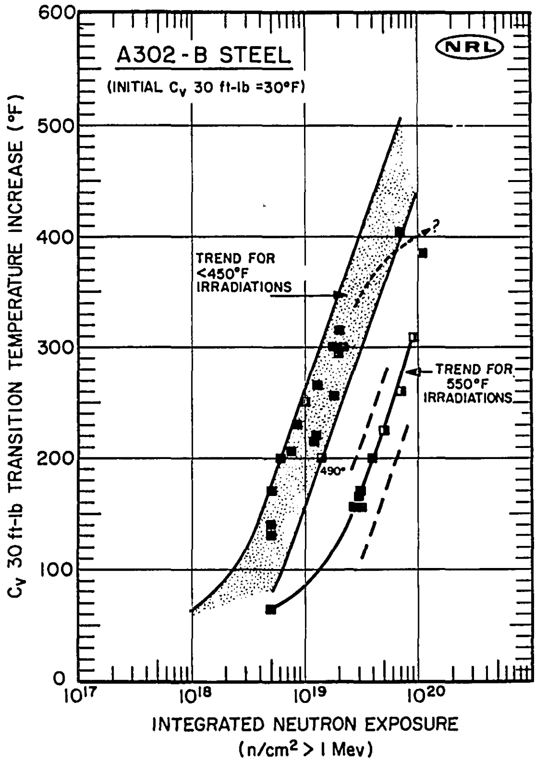

Fig. 1 — Irradiation embrittlement for ASTM 6-in. A302-B reference steel. The half-open symbols represent data obtained from irradiation in the Yankee Atomic Power Plant accelerated exposure surveillance capsule.

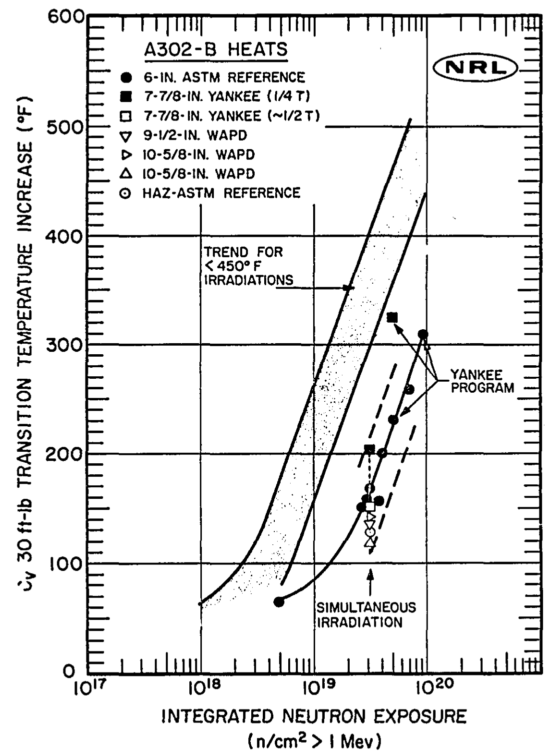

Fig. 2 — Data establishing the 550°F trend lines in Fig. 1. The irradiations were at or near 550°F in the Yankee Atomic Power Plant and in the Low Intensity Test Reactor. (WAPD indicates Westinghouse, Atomic Power Division.)

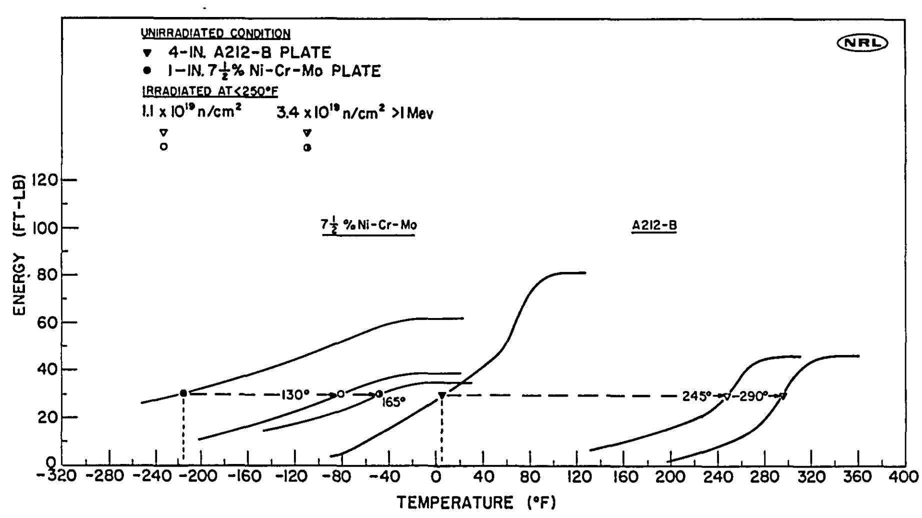

Fig. 3 — Charpy V-notch ductility characteristics of the high strength 7-1/2Ni-Cr-Mo steel relative to A212-B steel before and after irradiation to two neutron exposure levels at <250°F

which have shown trends quite similar to these. The dashed arrow suggest the beginning of embrittlement saturation of the A302-B reference steel. As a matter of background information, the basic trend band for <450°F irradiation was formed from the results of some sixty irradiation experiments involving ten different pressure vessel steels. Further, the width of the 550°F trend band was established by comparative irradiation of several heavy section heats of A302-B steel (Fig. 2) which were kindly furnished by representatives of the Westinghouse Electric Corporation, Atomic Power Division.

Low Temperature Irradiation Response

The first step in the comparative irradiation program involved capsule irradiation experiments conducted at temperatures below 280°F. The technique for comparing a reference steel and a higher strength steel is illustrated in Fig. 3, which shows the Charpy V-notch test results of simultaneous irradiation of the A212-B steel and an experimental steel, the 7-1/2Ni-Cr-Mo steel.

After simultaneous irradiation to two exposure levels, 1.1 and 3.4 × 10^{19} n/cm^2, the results of Charpy V-notch impact tests suggest the experimental high strength steel is much less sensitive to radiation embrittlement compared with the A212-B steel. Furthermore, the transition temperature increase for the high strength steel (165°F after an exposure of 3.4 × 10^{19}) results in a 30-ft-lb Charpy fix point* (−50°F) which is 55°F below the unirradiated *starting point* for the A212-B steel and 345°F below the as-irradiated transition temperature of the A212-B steel. Both steels showed a considerable drop in the full shear fracture absorption energy level, though that level for the higher strength steel was lower after irradiation than for the A212-B steel. Noteworthy, however, for both steels, are the relatively small increases in transition temperature for the 3.4 × 10^{19} exposure over that observed for the 1.1 × 10^{19} exposure—a marked indication of radiation damage saturation.

Additional data (Table 2) permit a comparison of results for several other steels which were exposed along with the A212-B and 7-1/2Ni-Cr-Mo steels. Of these several steels, only the experimental 7-1/2Ni-Cr-Mo steel and the SSS-100 steel showed significantly lower embrittlement. All the others show ΔT values at both irradiation levels which are so similar that no meaningful differentiation is possible.

The next step in the comparison involved irradiation of the A212-B and A302-B reference steels as well as the SSS-100 steel to a neutron dose of 1.1 × 10^{20} n/cm^2. Figure 4 compares the results obtained for the A302-B and SSS-100 steels and indicates a superior performance for the SSS-100 steel in terms of: the transition temperature increase (270 *versus* 385°F), the as-irradiated transition temperature (220 *versus* 420°F), and the as-irradiated full shear energy absorption level (53 *versus* 40 ft-lb). The potential advantage for a reactor pressure vessel steel having irradiated properties similar to those of the SSS-100 is readily apparent. The relatively low transition temperature after irradiation at low temperature to the very high exposure is especially encouraging. As illustrated in a later section, results for the SSS-100 steel after irradiation at 550°F are also very encouraging as are the results for elevated temperature irradiation of the two Ni-Cr-Mo steels.

When the notch ductility of the irradiated SSS-100 steel is compared for two dosage levels, 3.4 and 11.0 × 10^{19} n/cm^2, a very definite indication of embrittlement saturation is observed (Fig. 5). Although the full shear energy absorption value is lowered by a higher neutron dose, the transition temperature increase is only 15°F higher than that observed at only one-third the dose. A similar comparison for the A212-B and A302-B does not show such marked saturation. The latter steels show a ΔT value of ~300°F for the lower exposure and a ΔT of ~380°F for higher exposure. Thus, this comparison may suggest that saturation of the radiation embrittlement process occurs at a lower neutron

*The 30-ft-lb "fix point" position on the Charpy V-notch specimen curve is used to establish the transition temperature change induced by exposure to the high-energy nuclear radiation. The original basis for the use of this fix point was the correlation of the nil-ductility transition (NDT) temperature as established by the NRL drop-weight test with the Charpy curve for each steel evaluated. The 30-ft-lb level well represents the several A302-B steel heats studied and, while not specifically correlated for each of the higher strength steels evaluated in this report, is considered to be the best point for correlation of neutron induced increases for the purposes of this report. However, if the ΔT values generated in this study are to be used for assigning specific NDT values to the several steels, it would be necessary to use, in every case, the correlated NDT values which vary from about 30 ft-lb to over 70 ft-lb for the different high strength steels.

TABLE 2
Comparative Embrittlement of Several Steels
Irradiated Simultaneously at <280°F
(Based Upon Charpy V-Notch Tests)

Steel	Thickness (in.)	Initial C_V 30-ft-lb Value (°F)	Experiment A 1.1×10^{19} n/cm²		Experiment B 3.4×10^{19} n/cm²	
			Irrad. 30-ft-lb Value (°F)	ΔT (°F)	Irrad. 30-ft-lb Value (°F)	ΔT (°F)
A212-B*	4	5	250	245	295	290
A302-B	6	30	—	—	—	—
3-1/2Ni-Cr-Mo	8	−130	80	210	200	330
7-1/2Ni-Cr-Mo*	1	−215	−85	130	−50	165
A353	2	−370†	−125	245	−35	335
A387-B	6	30	—	—	345	315
A387-D	6	55	—	—	345	290
SSS-100	6	−45	—	—	210	255

*Shown in Fig. 3.
†Extrapolated value.

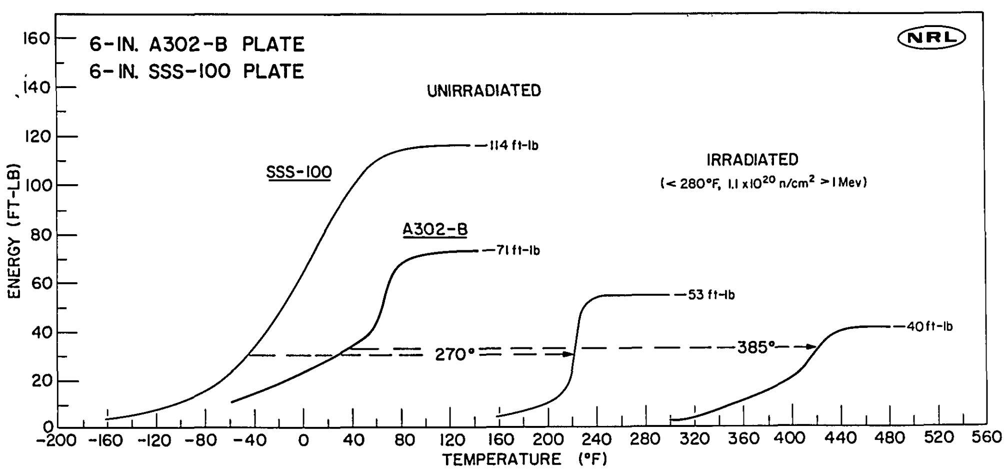

Fig. 4 — Relative Charpy V-notch ductility characteristics of SSS-100 and A302-B steels before and after exposure to a high neutron dosage (1.1×10^{20} n/cm²) at <280°F

dosage for the steel which shows lower sensitivity to embrittlement. However, much additional effort is required before this statement may be made without reservation.

Elevated Temperature Irradiation Response

For comparisons of embrittlement sensitivity with elevated temperature irradiations, 550°F was chosen as being most representative of reactor pressure vessel operating conditions. Space limitations reduced the number of steels which could be compared by means of simultaneous irradiation experiments. Nevertheless, the notch ductility behavior variations between the steels compared by this means were quite wide and were suggestive of the potential advantage for the higher strength steels.

This advantage, which can be measured in terms of both the low initial transition temperature

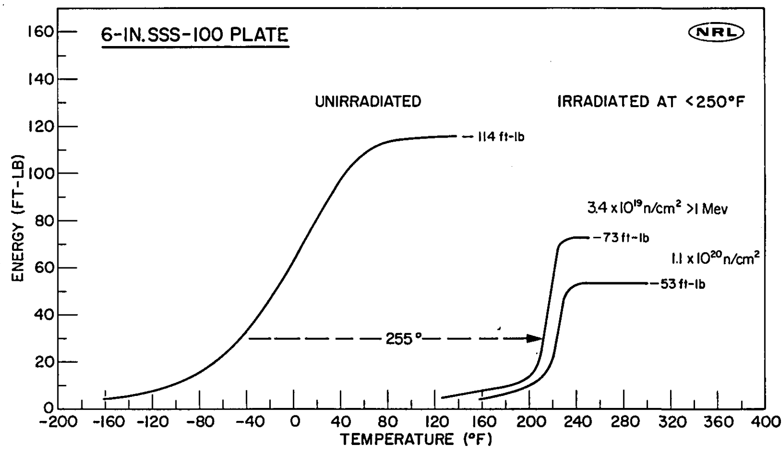

Fig. 5 — Charpy V-notch ductility characteristics of SSS-100 steel before and after irradiation to two exposure levels. The small additional increase for the higher exposure indicates marked embrittlement saturation.

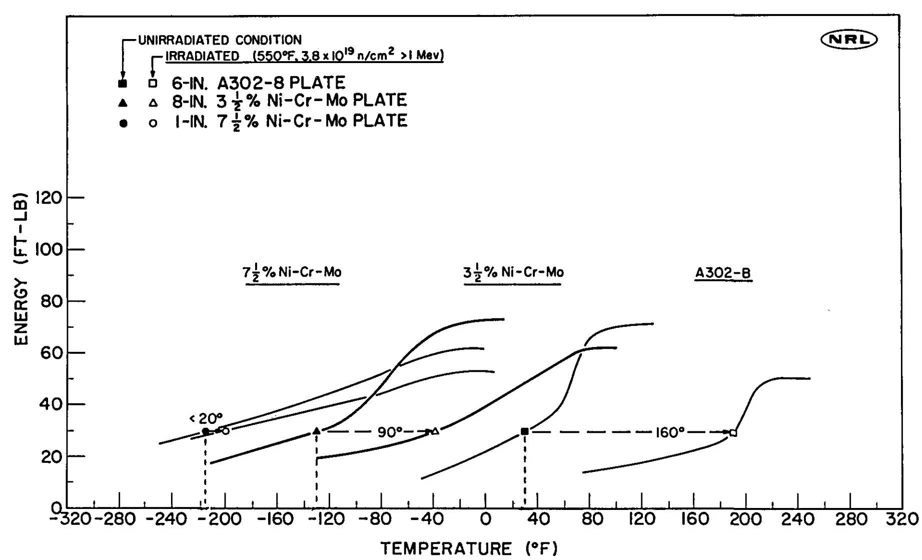

Fig. 6 — Charpy V-notch ductility characteristics of three steels of different strength levels after simultaneous irradiation at 550°F

and small sensitivity to radiation embrittlement, is demonstrated by comparing Charpy-V test results of three steels after irradiation (Fig. 6). The experimental 7-1/2Ni-Cr-Mo steel shows practically no increase in the transition temperature and relatively little drop in the full shear energy absorption level (62 to 53 ft-lb). The 3-1/2Ni-Cr-Mo steel, a commercial steel for which an ASME Code Case is being made, is somewhat more sensitive than the 7-1/2Ni-Cr-Mo steel but again shows much less transition temperature increase than the A302-B. Furthermore, the very low initial transition temperature of this steel coupled with the small radiation induced increase results in a transition temperature of −40°F after irradiation; that is, 70°F below the initial point for the unirradiated A302-B. When one considers that the exposure temperature well represents the operating temperature level of most commercial reactor vessels and that the exposure, 3.8×10^{19} n/cm^2,

TABLE 3
Charpy V-Notch Ductility Characteristics of Several Steels Irradiated Simultaneously at ~550°F* to 3.8 × 10^{19} n/cm² (>1 Mev)

Steel	Thickness (in.)	30-ft-lb Transition Temperature (°F)		
		Initial	Irradiated	ΔT
A212-B	4	5	190	185
A302-B†	6	30	190	160
3-1/2Ni-Cr-Mo†	8	−130	−40	90
7-1/2Ni-Cr-Mo†	1	−215	−200	15
A353	2	−370	−195	175
A387-B	6	30	190	160
A387-D	6	55	205	150

*The average specimen temperature reached 560°F for a significant portion of the irradiation period.
†Shown in Fig. 6.

TABLE 4
Charpy V-Notch Ductility Characteristics of 3-1/2Ni-Cr-Mo Steel (8-in. Plate) After Irradiation at 550, 700, and 750°F

Experiment	Irradiation Temperatures (°F)	Neutron Exposure (n/cm² >1 Mev)	30-ft-lb Transition Temperature (°F)		
			Initial	Irradiated	ΔT
A	550	1.5 × 10^{19}	−130	−30	100
B*	~550†	3.8 × 10^{19}	−130	−40	90
C	700	1.7 × 10^{19}	−130	−90	40
D	750	3.4 × 10^{19}	−130	15	145

*Shown in Fig. 6.
†The average specimen temperature reached 560°F for a significant portion of the irradiation period.

is greater than that expected for most reactor vessels, it is reasonable to assume that the pressure vessel embrittlement problem may be minimized if not eliminated in future reactors by careful selection of the vessel steel.

A more general comparison of the relative embrittlement between steels of various strength levels after irradiation at 550°F is shown in Table 3. It is apparent that, of the seven steels irradiated simultaneously in this experiment, only the two Ni-Cr-Mo steels show any appreciable advantage in terms of the degree of embrittlement, though it should be noted that the initial 30-ft-lb transition temperature for the A353 is quite low.

In a separate irradiation at 550°F to an exposure of 1.5 × 10^{19} n/cm², the SSS-100 showed an increase of only 110°F, suggesting a relatively small increase for this steel. Additional experiments are planned to better assess the threshold for embrittlement saturation for various steels irradiated at 550°F.

In drawing conclusions from the above data, it should be kept in mind that the data are limited, that an evaluation of the weld metal for these steels has not yet been completed, and that the propensity of this class of steels for radiation enhanced temper embrittlement has been suggested by earlier studies of a similar steel (2). These three deficiencies toward drawing conclusions will however be covered by studies now underway or planned for the near future.

In connection with temper embrittlement, some data are now available for the 3-1/2Ni-Cr-Mo after irradiation at 550, 700, and 750°F (Table 4). An indication that essentially complete saturation of neutron embrittlement occurs at or near an exposure of 1.5×10^{19} n/cm^2 is demonstrated by results for the two irradiations conducted at 550°F. With this fact in mind, the data for irradiation at 700 and at 750°F suggest a degree of temper embrittlement after 750°F irradiation. In view of the early saturation observed, the high exposure for the 750°F irradiation cannot fully explain the greater ΔT value (145°F) after exposure at that temperature. On the other hand, the low ΔT value (40°F) after irradiation at 700°F indicates that the increased embrittlement results primarily from irradiation at temperatures above 700°F. The combined effects of elevated temperature and neutron irradiation upon the degree of embrittlement of the several higher strength steels is being investigated further. Comparable out-of-reactor thermal aging experiments will be conducted also.

Directionality Considerations in Irradiated Steels

In the course of this comparative irradiation program it has been observed that the reduction in the full shear energy absorption level, especially at high neutron exposures, has been significant though not serious for the elevated temperature irradiation results. Thus, it is considered important to assess the potential fracture hazard presented by this factor in reference to possible directionality of properties in evaluating the higher strength steels.

In general, notch ductility evaluations of irradiated reactor pressure vessel steels have shown a continuous decrease in Charpy-V full shear energy absorption values with increasing neutron exposure. If the exposure is very large, the full shear energy may be decreased to a level approaching that for failure by low energy tear fracture (20 to 30 ft-lb) (5), and thus limit component serviceability, before extrapolations of transition temperature behavior from apparent trends would suggest a problem.

This possible limitation in serviceability becomes more evident when directionality in the properties of plate and forging materials are considered. In the process of comparing the properties of steels irradiated to very high exposure levels, an effort was made to assess the behavior of heavy section plates with respect to directionality. Charpy-V specimens of A212-B and A302-B steels, oriented with their long axis parallel (strong orientation) and transverse (weak orientation) to the plate primary rolling direction, were irradiated simultaneously at <280°F to a dosage comparable to the high dose on the SSS-100 steel (1.1×10^{20} n/cm^2 > 1 Mev).

Experimental results for the irradiated and unirradiated material conditions are presented in Figs. 7 and 8. Although having a nominal 1:1 cross rolling history, these steels in the unirradiated condition show a considerable difference in full shear energy absorption between their strong and weak directions (81 *versus* 63 ft-lb for the A212-B steel and 71 *versus* 46 ft-lb for the A302-B steel). With irradiation, an equivalent effect on both strong and weak orientations, in terms of the translation of the respective transition temperature curves to higher temperatures, was observed. While this observation would be in accordance with the predicted response, the data are highly significant in illustrating the possible need for assessing notch ductility in both the strong (normally specified) and weak plate directions. The full shear energy absorption values for both irradiated steels in the strong direction (43 ft-lb for the A212-B steel and 40 ft-lb for the A302-B steel) were above the critical range for failure by low energy tear fracture; however, properties in the transverse (weak) direction (25 and 18 ft-lb) would indicate susceptibility to ductile failure by this mode. Thus, while the results from specimens taken in the strong direction suggest transition temperature behavior as the limitation to be considered, the actual limitation in this case may be the poor full shear energy values developed in the weak or transverse direction.

Data for the postirradiated annealed condition are presented to demonstrate the possibility for restoring initial ductility by heat treatment.

The results obtained for the A212-B and A302-B steels in the weak direction after irradiation to 1.1×10^{20} suggest the need for more thorough evaluation of susceptibility to ductile failure in the weak direction in connection with the several high strengh steels studied. In this regard, specimens taken in the weak direction were chosen for irradiation evaluation of maraging and nickel-cobalt steels.

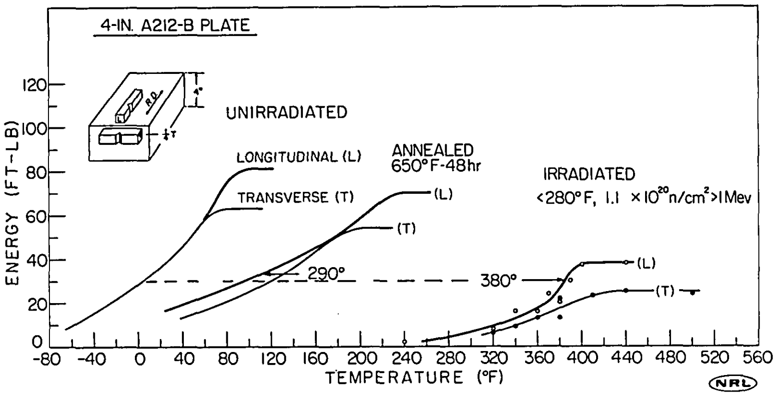

Fig. 7 — Charpy V-notch ductility characteristics of A212-B steel in the unirradiated, irradiated, and postirradiation annealed conditions showing the radiation response in the longitudinal and transverse directions

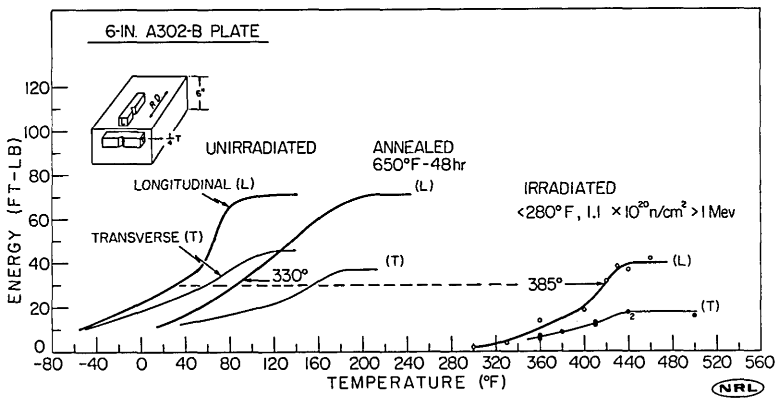

Fig. 8 — Charpy V-notch ductility characteristics of A302-B steel in the unirradiated, irradiated, and postirradiation annealed conditions showing the radiation response in the longitudinal and transverse directions

Characteristics of Irradiated Maraging and Nickel-Cobalt Steels—Preliminary Results

Certain maraging and nickel-cobalt bearing steels having yield strength levels in the range of 160 to 180 ksi are being studied as potential reactor structural materials. The steels chosen for investigation include seven heats of 12Ni-5Cr-3Mo maraging steels as well as a 9Ni-4Co-.25C steel in two cross rolling conditions. The melting practices employed for the two materials are vacuum induction melting and electric furnace air melting respectively, both followed by vacuum carbon deoxidation and vacuum consumable-electrode remelting. The composition and heat treatment of these steels is presented in Tables 5A and 5B and the mechanical properties before irradiation are given in Table 5C. The Charpy V-notch specimens were oriented in the weak direction (transverse to primary rolling direction) in order that these steels could be examined in the most unfavorable condition. Two steels, the 3-1/2Ni-Cr-Mo and 7-1/2Ni-Cr-Mo steels, were included along with the higher strength alloys for reference purposes. It should be noted, however, that the Charpy V-notch specimens of the reference steels were longitudinally oriented with respect to the primary rolling direction of the plates from which they were taken. The initial transition temperatures for the eleven steels compared are in the range −130 to −215°F.

TABLE 5A
Chemical Composition of 12-5-3 Maraging Steels (J1-J8) and 9Ni-4Co-.25C Steels (J14 and J15)

Steel	Chemical Analysis (%)										
	C	Mn	Si	P	S	Ni	Cr	Mo	Ti	Al	Co	V
J1	0.003	0.03	0.06	0.003	0.007	12.1	4.83	3.00	0.24	0.24	—	—
J3	0.003	0.03	0.06	0.003	0.007	12.1	4.96	3.10	0.24	0.24	—	—
J4	0.003	0.03	0.06	0.003	0.007	12.1	4.83	3.10	0.24	0.24	—	—
J5	0.003	0.03	0.06	0.003	0.007	12.1	4.83	3.10	0.24	0.21	—	—
J6	0.007	0.04	0.08	0.005	0.007	11.8	5.08	3.30	0.24	0.14	—	—
J7	0.005	0.03	0.06	0.003	0.007	12.1	4.83	3.10	0.24	0.22	—	—
J8	0.005	0.04	0.05	0.005	0.007	11.8	5.16	3.30	0.24	0.13	—	—
J14	0.25	0.29	0.01	0.004	0.008	8.62	0.40	0.48	—	—	3.76	0.11
J15	0.25	0.28	0.01	0.006	0.008	8.31	0.40	0.48	—	—	3.78	0.11

TABLE 5B
Heat Treatment of 12-5-3 Maraging Steels and 9Ni-4Co-.25C Steels

Steel	Heat Treatment
J1	Solution annealed at 1500°F for 2 hr; water quenched; aged at 900°F for 20 hr; water quenched.
J3	Solution annealed at 1500°F for 2 hr; water quenched; aged at 900°F for 2 hr; water quenched.
J4	Solution annealed at 1500°F for 2 hr; water quenched; aged at 900°F for 2 hr; water quenched.
J5	Solution annealed at 1500°F for 1 hr; water quenched; aged at 900°F for 20 hr; water quenched.
J6	Solution annealed at 1500°F for 1 hr; water quenched; aged at 900°F for 20 hr; water quenched.
J7	Solution annealed at 1500°F for 1 hr; water quenched; aged at 900°F for 20 hr; water quenched.
J8	Solution annealed at 1500°F for 1 hr; water quenched; aged at 900°F for 2 hr; water quenched.
J14	Normalized at 1600°F for 1 hr; air cooled; austenitized at 1500°F for 1 hr; oil quenched; tempered at 1000°F for 2 hr heating plus 2 hr holding; air cooled.
J15	Normalized at 1600°F for 1 hr; air cooled; austenitized at 1500°F for 1 hr; oil quenched; tempered at 1000°F for 2 hr heating plus 2 hr holding; air cooled.

TABLE 5C
Mechanical Properties of 12-5-3 Maraging Steels, 9Ni-4Co-.25C Steels, and (For Reference Purposes) Two Ni-Cr-Mo Steels

Steel	Charpy-V 30-ft-lb Transition Temp. (°F)	Yield Strength (0.2% Offset) (ksi)	Tensile Strength (ksi)	Elongation (1-in. gage length) (%)	Reduction of Area (%)
J1	−180	179.1	185.1	15.5	64.5
J3	−200	170.6	175.6	16.0	66.0
J4	−160	162.8	170.9	16.6	64.4
J5	−165	176.2	185.3	13.5	58.3
J6	−175	184.4	190.0	13.5	55.7
J7	−180	181.5	188.5	14.5	62.3
J8	−200	162.6	170.6	15.8	64.1
J14	−135*	180.3	196.4	15.0	48.0
J15	−135	183.2	195.0	17.0	61.0
3-1/2 Ni-Cr-Mo	−130	95.0	122.0	24.7	68.7
7-1/2 Ni-Cr-Mo	−215	148.8	169.4	19.0	68.7

*Based on Charpy-V 25-ft-lb level.

Specimens were irradiated in the Union Carbide Research Reactor (UCRR) at <250°F to an exposure of ~7 × 10^{18} n/cm² (>1 Mev) under a partial dry-helium atmosphere in stainless steel encapsulated assemblies. The relatively low dosage was used in order to promote a rapid preliminary examination of properties and to facilitate early elimination of any unlikely candidate steels.

The postirradiation notch ductility characteristics for two selected maraging steels representing the low and high ends of the yield strength range are presented in Figs. 9 and 10. A summary of the properties of the eleven irradiated steels included in this study is presented in Table 6.

In general, the 12Ni maraging steels demonstrated transition temperature increases of ~30 to ~50°F and a lowering of the full shear energy absorption level of about 5 to 20 ft-lb for an exposure of ~7 × 10^{18} n/cm² (>1 Mev). No significant difference in irradiation response can be assigned to the various heat treatment conditions of the seven plates (Table 5B).

As the cobalt-bearing steels exhibited fairly low initial Charpy V-notch full shear energy absorption levels (30 to 40 ft-lb), it was necessary to use a 25-ft-lb-level criterion to assess irradiation induced increases in transition temperature. At the 25-ft-lb level the transition temperature increases were very small (20 to 25°F), and the full shear energy absorption values were depressed somewhat (<10 ft-lb) below the initial low values. In contrast to the behavior of the maraging steels, the very low full shear energy absorption values and very high induced radioactivity of the cobalt-bearing steel effectively eliminates it from serious consideration for nuclear structural application.

In comparison with the above results for the 160 to 180 ksi yield strength steels, the 7-1/2Ni-Cr-Mo control steel showed an initial transition temperature of −215°F and an increase due to irradiation of 45°F, while the corresponding values for the 3-1/2Ni-Cr-Mo control steel were −130 and 135°F. In general, the properties of the irradiated maraging steels correspond favorably with those of the 7-1/2Ni-Cr-Mo steel. And, like this experimental steel, the maraging steels proved to be rather insensitive to neutron induced radiation damage. The favorable initial results have stimulated further study of the maraging steels to determine the tensile properties after irradiation as well as to assess the effects of higher neutron exposure and elevated temperature irradiation upon the notch ductility characteristics.

SUMMARY AND CONCLUSIONS

Although the scope of this initial comparative irradiation embrittlement study of higher strength steels was necessarily limited, it has achieved the exploratory assessment of relative irradiation behavior of steels of several types and strength levels. These include lower strength Cr-Mo steels,

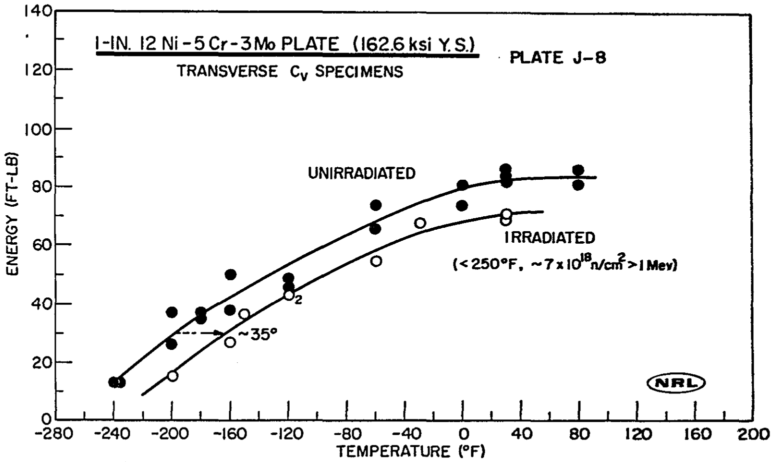

Fig. 9 — Charpy V-notch ductility characteristics of a 12Ni-5Cr-3Mo maraging steel before and after irradiation at <250°F. This steel represents the lower portion of the strength range for the group of maraging steels studied.

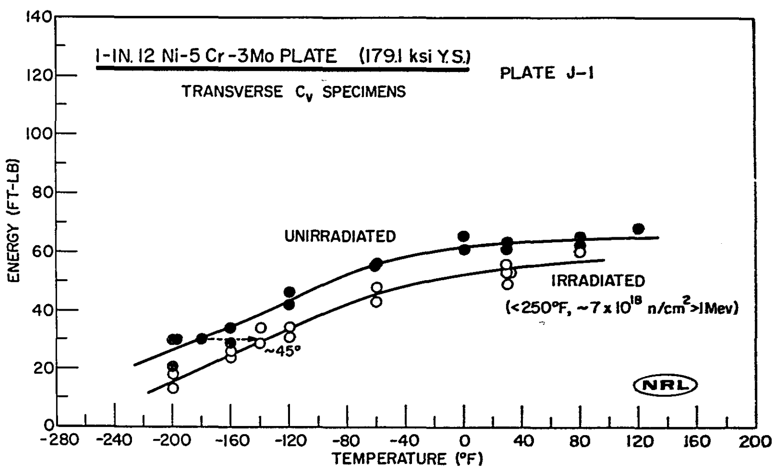

Fig. 10 — Charpy V-notch ductility characteristics of a 12Ni-5Cr-3Mo maraging steel before and after irradiation at <250°F. This steel represents the higher portion of the strength range for the group of maraging steels studied.

medium strength Ni-Cr-Mo steels, and a group of the high strength maraging and nickel-cobalt steels which together represent a fairly good cross section of recently developed steels. The irradiation conditions, temperature, and neutron dose, while not all encompassing, permitted a general comparison of the properties of the several higher strength steels relative to those of A212-B and A302-B steels after irradiation.

From these comparisons, certain conclusions of potentially great significance to the future application of advanced steels for reactor pressure containment applications can be tentatively advanced. These conclusions are stated with reference to a summary graph (Fig. 11) which compares the initial and neutron induced transition temperatures (NDT) for steels of selected strength levels. It was considered beneficial to add the A350-LF3* steel to this graphic presentation because of its greater sensitivity to neutron

*The A350-LF3 forged steel (representative of the PM-2A reactor pressure vessel) has a yield strength of 59 ksi and chemical composition (weight-percent) as follows: 0-14C, 0.25Si, 0.031P, 0.32S, 3.28Ni, and 0.52Mn.

TABLE 6
Charpy V-Notch Ductility Characteristics of 12-5-3 Maraging Steel (J1-J8), 9Ni-4Co-.25C Steel (J14 and J15), and (For Reference Purposes) 3-1/2Ni-Cr-Mo and 7-1/2Ni-Cr-Mo Steels After Irradiation at ~7 × 10^{18} n/cm² (>1 Mev) at <250°F

Steel	30-ft-lb Transition Temperature (°F)			Full Shear Energy Absorption	
	Initial	Irradiated	ΔT	Average Initial (ft-lb)	Irradiated Reduction Δ (ft-lb)
J1*	−180	−135	45	65	<10
J3	−200	−145	55	75	<10
J4	−160	−135	25	75	<10
J5	−165	−115	50	70	<20
J6	−175	−125	50	65	<10
J7	−180	−150	30	65	<10
J8†	−200	−165	35	80	<10
J14	−135	−110	25‡	35	< 5
J15	−180	−160	20‡	45	<10
3-1/2Ni-Cr-Mo	−130	5	135	70	<20
7-1/2Ni-Cr-Mo	−215	−170	45	65	<10

*Shown in Fig. 10.
†Shown in Fig. 9.
‡Based on Charpy-V 25-ft-lb level.

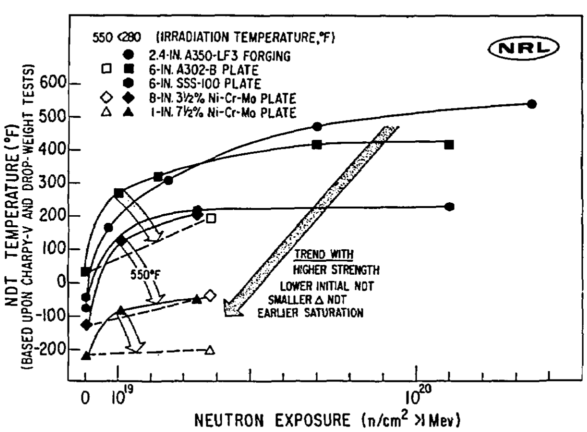

Fig. 11 – The relative nil-ductility transition (NDT) temperature values for five steels having yield strength levels from about 60 to 180 ksi compared before irradiation (data points at zero on the abscissa, which is linear) and after irradiation at <280°F and at 550°F. The dashed lines represent approximate trends only.

embrittlement and the availability of data to very high neutron dosage levels.

Figure 11 presents NDT temperature values (based upon Charpy-V 30-ft-lb levels) both before and after irradiation for five selected steels representing various strength levels from the A350-LF3, having a yield strength of 59 ksi, to the 7-1/2Ni-Cr-Mo, having a yield strength of 148 ksi.* Except in the case of the A350-LF3, the data points are those representing the simultaneous irradiation experiments discussed in this report.

The primary observations which may be derived from examination of Fig. 11 include the following:

1. The higher strength steels exhibit a general trend toward lower initial NDT indicated by the range from A302-B (70-ksi yield strength) at +30°F to the 7-1/2Ni-Cr-Mo (148-ksi yield strength) at −215°F.

2. Along with higher strength and lower initial NDT, there is also a tendency for smaller radiation induced increases in transition temperature (ΔNDT) as indicated by the relative positions of the five curves.

3. There appears to be a general tendency for earlier saturation of radiation embrittlement with increasing strengths. With the A350 steel, complete saturation is not observed even at the exposure level of 1.35 × 10^{20}, whereas with

*For perspective, it should be noted that the limited results for the maraging steels (160 to 180 ksi yield strength) indicate a behavior quite similar to that observed for the 7-1/2-Ni-Cr-Mo steel (see Table 6).

the 7-1/2Ni-Cr-Mo there is a strong indication of saturation in the exposure region between 1.1 and 3.4×10^{19} n/cm^2. The data for the other steels give indications of saturation at intermediate exposure levels—approximately 7×10^{19} n/cm^2 for the A302-B and approximately 3×10^{19} n/cm^2 for the SSS-100 steel.

4. A marked and progressive advantage in terms of lower radiation embrittlement is observed with the higher strength steels irradiated at 550°F. The benefit of 550°F irradiation is particularly apparent in the case of the Ni-Cr-Mo steels, the increase for the 3-1/2Ni-Cr-Mo steel being only 90°F and for the experimental 7-1/2 Ni-Cr-Mo steel only 15°F compared with an increase for the A302-B at the same exposure (3.8×10^{19} n/cm^2) of 160°F.

5. Though limited, the data for irradiation at 550°F suggest that even earlier saturation of radiation embrittlement is obtained for the higher strength steels after elevated temperature exposure; however, additional data are required to fully clarify this point.

The very low initial NDT of the higher strength steels coupled with the smaller ΔNDT and earlier apparent saturation of irradiation embrittlement would suggest that the use of steels having properties similar to those presented in this report may effectively eliminate any major concern for radiation embrittlement of future reactor pressure vessel steels. This suggestion is vividly demonstrated by the fact that the 3-1/2Ni-Cr-Mo steel for which an ASME Code Case (No. 1358) has been made, exhibits an NDT of −40°F after an exposure of 3.8×10^{19} n/cm^2 at 550°F compared with an NDT for the A302-B steel under the same exposure conditions of 190°F. Further, the transition temperature for the Ni-Cr-Mo steel after irradiation is 70°F below the transition temperature for the A302-B steel *before* irradiation. Thus, while one might have to consider radiation embrittlement at this exposure level for the A302-B steel, it would not be a consideration for the 3-1/2Ni-Cr-Mo steel.

Of course, these conclusions must be stated with full cognizance of certain limitations to the data available at this time. One of these limitations is the question of the reduction of the full shear energy absorption level on the Charpy-V curve after irradiation. Results for the 550°F irradiation would suggest that this is not a problem with the steels studied. Nevertheless, this possibility must be considered. Another question is the effect of irradiation on the weld metals and weld heat-affected zone of the higher strength steels. An investigation is required to determine whether the advantages outlined for the higher strength steels after irradiation hold also for the weld and weld heat-affected zone of these same steels. A further question relates to the possibility of radiation induced temper embrittlement for the higher strength steels, a question which can only be resolved by long term elevated temperature exposures. From experimental results obtained to date, however, this might prove to be a problem only at temperatures above 700°F.

In summary, then, it may be concluded from this study that with irradiation several steels having higher strength and low initial NDT exhibit small increases in NDT and early saturation of irradiation embrittlement and that with elevated temperature irradiation they exhibit very small NDT increases. Thus, these steels have many favorable features for structural use in future reactor systems.

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REFERENCES

1. Rice, W.L.R., "Irradiation Capsule Experiments," TID-7697, U.S. Atomic Energy Commission, 1964
2. Steele, L.E., and Hawthorne, J.R., "New Information on Neutron Embrittlement and Embrittlement Relief of Reactor Pressure Vessel Steels," Symposium on Flow and Fracture Behavior of Metals and Alloys in Nuclear Environments, ASTM STP 380, *Am. Soc. Testing Matls.*, Philadelphia, 1965, pp. 283-311
3. Steele, L.E., and Hawthorne, J.R., "Neutron Embrittlement of Reactor Pressure Vessel Steels," pp. 366-409 in "Materials

and Fuels for High-Temperature Nuclear Energy Applications," Cambridge, Mass.:MIT Technology Press, 1964
4. Alger, J.F., and Porter, L.F., "Evaluation of Reference Pressure-Vessel Steels for Neutron-Irradiation Studies," U.S. Steel Applied Research Laboratory, June 18, 1964
5. Pellini, W.S., and Puzak, P.P., "Practical Considerations in Applying Laboratory Fracture Test Criteria to the Fracture-Safe Design of Pressure Vessels," NRL Report 6030, Nov. 5, 1963

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13. ABSTRACT

Several steels representative of recently developed types and having potential for nuclear structural applications were exposed to high energy nuclear radiation, and the resultant properties were compared with those of the currently used A212-B and A302-B nuclear reactor pressure vessel steels. Preliminary results from several comparative irradiation experiments indicate that certain higher strength steels, in addition to having initial qualities of higher strength and lower initial ductile-brittle transition temperatures, show smaller embrittlement, earlier embrittlement saturation, and a superior overall response to irradiation at 550°F than that observed for the steels in current reactor pressure vessels.

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	ROLE	WT	ROLE	WT	ROLE	WT
Steels High Strength Steels Maraging Steels Ductile-Brittle Transition Temperature Nil Ductility Transition (NDT) Temperature Fracture Energy Pressure Vessel Pressurized Water Reactors Nuclear Reactor Containment Neutron Exposure Embrittlement Irradiation Embrittlement Neutron Embrittlement Neutron Embrittlement Sensitivity						

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