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Irradiation Effects on Reactor Structural Materials

Quarterly Progress Report - 1 May - 31 July 1967

August 15, 1967

NAVAL RESEARCH LABORATORY
Washington, D.C.

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ABSTRACT

The research program of the NRL Metallurgy Division, Reactor Materials Branch, is devoted to the determination of the effects of nuclear radiation upon the properties of structural materials. The overall program is sponsored by the Office of Naval Research, the Navy Ship Systems Command, the U. S. Atomic Energy Commission, and the Army Nuclear Power Program. Since research findings which apply to the objectives of one sponsoring agency are also of interest to the others, the overall program progress is reported herein. This report, covering research for the period 1 May-31 July 1967, includes the following: (1) through-thickness radiation embrittlement sensitivity of two A533 Grade B, Class I steel plates at 550°F, (2) directional notch ductility performance of irradiated 3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V steel plates, (3) radiation sensitivity of A353 (9% nickel) steel as influenced by percent retained austenite, (4) tensile properties behavior versus postirradiation test temperature of selected structural steels, (5) potential for aging embrittlement of pressure vessel steels, (6) postpressurization test operations on PM-2A reactor pressure vessel, and (7) auxiliary equipment developed for elevated temperature remote tension testing of radioactive specimens.

PROBLEM STATUS

This is a quarterly progress report; work on this problem is continuing.

AUTHORIZATION

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Editor Note: This report will be included as a part of the quarterly progress report which is compiled for the AEC and published by the Battelle-Northwest Laboratory, Richland, Washington, on this subject. Figures and Tables are identified as Section 8 in conformance with the Battelle publication.

SUMMARY

RADIATION EMBRITTLEMENT SENSITIVITY OF TWO A533 GRADE B, CLASS I STEEL PLATES

Charpy V-notch specimens of 4- and 8-in. production plates from one heat of nickel bearing A533 Grade B, Class I, steel were irradiated simultaneously at a controlled temperature of 550°F to a neutron fluence of 2.3×10^{19} n/cm^2 >1 Mev. Specimen groups representative of surface, quarter thickness, and half thickness locations of each plate were evaluated. Neither the 4- or 8-in. thick plate showed through-the-thickness variations in radiation embrittlement sensitivity although differences in preirradiation transition temperatures were observed from plate surface to center. The transition temperatures of both A533 plates after irradiation were only slightly higher than room temperature while that of a 6-in. reference A302-B plate included in the experiment was close to boiling water temperature.

NOTCH DUCTILITY BEHAVIOR OF IRRADIATED 3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V STEEL PLATE BOTH PARALLEL AND TRANSVERSE TO THE PRIMARY ROLLING DIRECTION

Charpy-V notch ductility properties in the strong (primary rolling direction) and weak plate orientations of two quenched and tempered steels, 3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V, have been assessed and compared after irradiation at <250°F. Individual neutron fluence values were 1.6×10^{19} n/cm^2 >1 Mev and 5.5×10^{18} n/cm^2 >1 Mev respectively. Each steel exhibited an equivalent transition temperature increase in its strong versus weak plate orientation. The data also suggested comparable losses in full shear energy absorption levels for the two plate orientations by irradiation. This observation was consistent with the behavior noted previously for the ASTM A212-B and A302-B reference steel plates for the <250°F exposure condition.

RADIATION SENSITIVITY OF A353 (9% NICKEL) STEEL CONTAINING VARIOUS AMOUNTS OF RETAINED AUSTENITE

The effect of high neutron exposure on the impact and tensile properties of A353 steel containing various amounts of retained austenite has been investigated in two low temperature irradiation (<250°F) experiments. Stable retained austenite in the amounts of 3.3, 7.1, 7.2, and 11.2 percent as measured by X-ray diffraction was initially produced in 1-in. quenched and tempered A353 plate by retempering the material in the range of 1050 to 1100°F. Specimens from commercially heat treated 1-in. and 2-in. A353 steel plates selected for reference were irradiated with the experimentally heat treated material. Charpy

V-notch specimens were irradiated in the Materials Test Reactor (MTR) to a neutron fluence of 7.3×10^{19} n/cm^2 >1 Mev, while tensile specimens were irradiated in the Low Intensity Test Reactor (LITR) to 6.9×10^{19} n/cm^2 >1 Mev.

The test results did not indicate any precise trends in irradiation response as a function of retained austenite content, although differences were noted among the four experimental test plates. The two commercially heat treated A353 reference plates showed a somewhat higher NDT temperature increase than the experimental test plates, but the observed differences were not sufficient to form positive conclusions. The tensile data similarly did not reveal a definite correlation between the amount of retained austenite and radiation sensitivity of A353 steel.

TENSILE PROPERTIES VERSUS TEST TEMPERATURE FOR IRRADIATED STRUCTURAL STEELS

The stress-strain responses of seven steels that were post-irradiation tested extensively at ambient temperature (~75°F) have now been determined to temperatures up to 750°F. Testing has been completed for specimens exposed respectively to 0.85×10^{19}, 2.3×10^{19}, and 9.5×10^{19} n/cm^2 >1 Mev for each of the seven steels. Most testing was concentrated at the three temperatures 450, 550, and 650°F which constitute projected minimum, intermediate, and maximum temperatures for nuclear pressure vessel operation. To facilitate construction of the temperature-property curves, selected specimens of A212-B and A302-B steel were also tested in the unirradiated condition at temperatures from 250 to 750°F.

AGING EMBRITTLEMENT OF PRESSURE VESSEL STEELS

A preliminary study has been undertaken to assess the susceptibility of A302-B steel to aging embrittlement. Emphasis has been placed on typical water cooled reactor pressure vessel operations as providing conditions which may be favorable for the development of this form of embrittlement.

A fracture mechanics investigation was conducted below the nil ductility transition (NDT) temperature of an A302-B steel test plate using specimens that were preloaded and later aged at 550°F. Reference specimens were also preloaded but were not aged at temperature. The investigation indicated some embrittling effect as noted by a 23 percent lower K_{Ic} value for aged versus unaged specimens at -250°F. The results point to a need for further definition of this phenomenon including studies which couple irradiation and aging treatments.

POST-PRESSURIZATION TEST OPERATIONS ON PM-2A REACTOR PRESSURE VESSEL

The Naval Research Laboratory has been assigned the responsibility for coordinating efforts to obtain and test metallurgical specimens from the PM-2A reactor pressure vessel as required by the third (post-pressurization) phase of planned vessel tests. Progress on this effort to date is reported.

GAGE MARKING DEVICE FOR IRRADIATED TENSILE SPECIMENS

In testing radioactive tensile specimens at elevated temperatures it was discovered that failure often initiated at the indentations introduced to establish the initial 1-in. gage length. To eliminate this problem a technique for etching the 1-in. gage length reference marks was developed and placed into operation in the tension testing facility of the High Level Radiation Laboratory.

RESEARCH PROGRESS

Radiation Embrittlement Sensitivity of Two A533 Grade B, Class I, Steel Plates

The use of A302-Grade C and A533-Grade B pressure vessel steels is now planned for several new, pressurized and boiling water reactor systems. The effects of the nickel modification on the general radiation embrittlement sensitivity of plate material, therefore, requires investigation and assessment based on the known response of the non-modified A302-Grade B composition. Further, the possibilities of a variation in embrittlement sensitivity with thickness layer through heavy section plate similarly requires exploration since such a variation has been observed in previous studies on conventional A302-Grade B plate(1). For best representation of projected pressure vessel service conditions, an irradiation temperature of 550°F is probably most appropriate for experimental tests.

Charpy V-notch specimens of both 4- and 8-in. plate forms of one heat of nickel bearing A533-Grade B, Class I, steel (Table 8.1) were irradiated simultaneously at a controlled temperature of 550°F in the Oak Ridge Low Intensity Test Reactor. The neutron fluence received was 2.3×10^{19} n/cm^2 >1 Mev as determined from iron dosimeter wires included in the assembly. To allow experimental through-thickness determinations of relative irradiation response, specimen groups representative of surface, quarter thickness, and half thickness locations of each plate were included in the irradiation assembly. In addition, the experiment provided for a single set of control test specimens taken from the quarter thickness location of a 6-in. plate of conventional A302-Grade B steel (the ASTM reference steel plate).

Experimental results obtained for the A533-Grade B composition are given in Figs. 8.1 and 8.2. On comparing data pertaining to a single plate, the transition temperature increases for the three thickness locations do not reveal any difference in irradiation response from plate surface to center thickness positions. The marked difference in the preirradiation properties noted between the surface and quarter thickness regions of the 4-in. plate is not surprising in view of the relatively thin section size involved versus the hardenability characteristics of this composition. Thus, it is particularly significant that these two thickness locations responded equally to the radiation exposure.

From Figs. 8.1 and 8.2 it is seen that the general irradiation responses of the 4- and 8-in. plates were about equal.

Moreover, the final transition temperatures of both plates after irradiation were only slightly higher than room temperature. In contrast to this behavior, the ASTM reference steel exhibited a transition temperature increase of 180 degrees and a final, as-irradiated transition temperature close to boiling water temperature. Assuming that the Charpy-V 30 ft-lb temperature is a reasonable index of the NDT temperature of this steel, and that NDT plus 60 degrees is the criterion for minimum temperature for full reactor pressurization during plant startup (or shutdown), a superiority of nickel bearing A533-Grade B steel over conventional A302-Grade B steel is suggested.

It is planned that additional heats of A533-Grade B steel will be examined in the interest of confirming both a general absence of through-thickness gradients in radiation embrittlement sensitivity and a material trend toward lower radiation embrittlement relative to the non-nickel modified A302-Grade B steel.

<u>Notch Ductility Behavior of Irradiated 3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V Steel Plate Both Parallel and Transverse to the Primary Rolling Direction</u>

It has become increasingly apparent that consideration should be given to the loss in full shear energy absorption of steels as well as to the transition temperature increase produced by neutron radiation. This would appear to be particularly important in evaluating the performance of the newer quenched and tempered pressure vessel steels which have much lower initial transition temperatures than the A212-B and A302-B steels now in wide usage. In an earlier report (2), it was demonstrated that, even for these conventional steels, the loss in full shear energy absorption as measured for the transverse (i.e., weak) plate orientation may be such (20/30 ft-lbs) as to limit serviceability before the transition temperature increase would become an operational limitation. Investigations of the relative irradiation effects on directional notch ductility behavior have therefore continued in the interest of establishing trends in performance for various test orientations.

Charpy V-notch specimens taken parallel (strong orientation) and transverse (weak orientation) to the primary rolling direction of 8-in. 3-1/2Ni-Cr-Mo plate and of 1-in. 5Ni-Cr-Mo-V plate have been irradiated at <250°F in separate experiments to neutron fluences of 1.6×10^{19} n/cm^2 >1 Mev and 5.5×10^{18} n/cm^2 >1 Mev respectively. The chemical composition and heat treatment of the test plates are given in Table 8.2 Table 8.3 summarizes changes found for both plate orientations with respect to transition temperature increase and loss in full shear energy absorption. The comparison of transition temperature increases noted for an individual steel indicates no significant effect of plate orientation on apparent radiation embrittlement sensitivity, Similarly, the average loss in full shear energy absorption is found to be independent of plate orientation for both steels. Important to this particular study, the average full shear energy absorption level of the 3-1/2Ni-Cr-Mo steel plate (transverse orientation) was reduced by irradiation from 59 to 41 ft-lbs while a transition temperature just slightly above room temperature was recorded with specimens conventionally oriented in the direction of primary rolling. In the case of the 1-in. 5Ni-Cr-Mo-V plate, significant notch toughness was retained in both orientations as would be expected for its low fluence condition.

Studies were also conducted on postirradiation annealing response of the 3-1/2Ni-Cr-Mo steel as a function of test orientation. The data suggested parallel recovery in transition temperature following 650°F and 750°F, 168-hour heat treatments. Although limited, the 750°F annealing data further indicated equivalent response toward full shear energy absorption recovery.

Summarizing, the data presented in Table 8.2, together with that given earlier for A212-B and A302-B, are in agreement in suggesting that the preirradiation difference in average full shear energy absorption in the strong versus weak plate orientations is maintained with irradiation. Preirradiation differences in transition temperatures also appear to be maintained with irradiation. Thus, the data signify that, depending upon the particular steel involved, either a reduced full shear energy absorption level or an increased transition temperature may ultimately become the controlling factor determining continuing component serviceability.

<u>Radiation Sensitivity of A353 (9% Nickel) Steel Containing Various Amounts of Retained Austenite</u>

The irradiation behavior of A353 steel with varying amounts of retained austenite has been investigated in two low temperature irradiation (<250°F) experiments. A353 steel is attractive for reactor structural applications (liners, containment vessels) because of its very low initial NDT temperature and its relatively high strength. It has been suggested that increasing the austenite content to a higher level than that which is generally developed by commercial heat treatment may increase the resistance of this material to irradiation damage.

Large amounts of austenite can be developed in this material by varying the heat treatment. Austenite thus formed is stable at room temperature, but only a portion of the austenite is stable at sub-zero temperatures. The aim of this investigation was to evaluate the resistance to radiation damage of A353 material containing 3 to 15 percent stable austenite developed by re-tempering quenched and tempered plate in the range of 1050 to 1100°F. A list of materials used in this study, together with their chemical composition, heat treatment, and the amount of retained austenite developed by each treatment is given in Table 8.4. Specimens from two commercially heat treated plates - double normalized and tempered 1-in. plate and quenched and tempered 2-in. plate - were included in this investigation for reference. The austenite contents given in Table 8.4 for the unirradiated experimental plates were determined to be stable over the Charpy-V testing temperature range used in this experiment.

Charpy-V specimens of each material were irradiated simultaneously in the MTR at <250°F to a neutron fluence of 7.3 x 10^{19} n/cm^2 >1 Mev. A comparison between the unirradiated and irradiated notch impact properties for all materials is made in Table 8.5 and presented graphically in Figs. 8.3 through 8.5. The impact test results do not indicate any precise trends in irradiation response as a function of retained austenite contents. The two commercial heat treated A353 plates show somewhat higher NDT temperature shifts than the experimental test plates, but the observed differences are not clearly significant.

Charpy-V data curves for the unirradiated condition of the experimental plates show a dip or point of inflection near the upper energy shelf which is not evident in the commercially heat treated plates. The reason for, or the significance of, this behavior has not been established. Figures 8.3 through 8.5 also indicate a slight decrease in the slope or flattening of the unirradiated Charpy energy curves with increasing retained austenite content resulting in appreciably lower initial NDT temperature for plate having the 11.2 percent retained austenite content. The NDT temperature increase for this plate following irradiation, however, was comparable to plates containing lesser amounts of retained austenite. Experimental plates R2 and R3, both containing nominally 7 percent retained austenite, were tempered at different temperatures prior to irradiation. Plate R2 received a 60-hour treatment at 1050°F resulting in 7.1 percent retained austenite while plate R3 was tempered 7 hours at 1100°F to produce 7.2 percent retained austenite. The Charpy-V transition temperature behavior of these plates, however, does not suggest any significant differences in irradiation response.

Tensile specimens of the steel series were irradiated simultaneously in the LITR at <250°F to a neutron fluence of 6.9×10^{19} n/cm^2 >1 Mev. The unirradiated and irradiated properties of the materials are summarized in Table 8.6. As in the case of the ductile-brittle transition temperature behavior, the tensile data do not suggest a trend in irradiation response as a function of retained austenite. In comparing the two experimental plates, R2 and R3, described in the previous paragraph, it appears that Plate R3 (1100°F temper) experienced a significantly smaller change in tensile strength than Plate R2 (1050°F temper). On the basis of yield strength, however, both plates behaved quite similarly.

In summary, it can be concluded that varying the amount of stable retained austenite in A353 steel through a range of 3 to 11 percent does not significantly affect the irradiation response of this material as measured by changes in tensile and impact behavior. This statement applies for low temperature (<250°F) irradiation to a high neutron fluence (7×10^{19} n/cm^2 >1 Mev).

Tensile Properties Versus Test Temperature for Irradiated Structural Steels

The stress-strain responses of seven steels that were postirradiation tested extensively at ambient temperature (~75°F) for possible application in a nuclear environment (3) have now been determined to temperatures as high as 750°F. Most testing, however, has been concentrated at the three temperatures 450, 550, and 650°F, which constitute projected minimum, intermediate, and maximum temperatures for nuclear pressure vessel operation. To facilitate construction of the temperature-property curves, selected specimens of A212-B and A302-B steel were also tested in the unirradiated conditions at temperatures from 250 to 750°F. Testing has been completed for specimens exposed respectively to 0.85×10^{19}, 2.3×10^{19}, and 9.5×10^{19} n/cm^2 >1 Mev for each of the seven steels.

The yield and tensile strengths as functions of temperature for the unirradiated reference steels are presented in Fig. 8.6. The yield strengths for both steels are reduced by nearly 10 ksi as the testing temperature is raised from 75 to 350°F. As the testing temperature is further increased to 650°F, the yield strength remains constant for the A212-B steel but then drops with a further temperature increase. For the A302-B steel, on the other hand, the yield strength increases with increasing temperature above 350°F to develop a secondary maximum at 550°F. As the testing temperature is increased to 650°F and then to 750°F, a reduction in the yield strength is clearly developed.

The tensile strength curves for the two steels show well developed secondary maxima at from 550 to 600°F which corresponds to the conventional "500-degree embrittlement" temperature range. For both steels the minimum tensile strengths measured at 350 to 450°F are about 10 ksi less than 75°F values, while the secondary maximum values are about equal to the 75°F values.

The 500-degree embrittlement behavior observed for the unirradiated condition is also indicated for A212-B steel at all irradiation fluences studied, as can be seen in Fig. 8.7. For A302-B steel (Fig. 8.8), the 500-degree embrittlement effect is well established for the minimum and intermediate fluence levels but is not evident after exposure to 9.5×10^{19} n/cm^2 >1 Mev.

Tentatively, 500-degree embrittlement has been observed for A212-B, A302-B, A350-LF3, and A353 steel. For steels T-1, A350-LF1, and HY-80, 500-degree embrittlement was not clearly evident from the strength-temperature curves, although it is emphasized the data for the unembrittled conditions are not yet available for these steels.

In this preliminary analysis of the temperature dependence of stress-strain behavior of the steels in the irradiation study, the data are reported as conventional tensile and yield strengths, percent reduction of area, $(1 - \sigma_{YS}/\sigma_{TS})$, and δ_{ML}, the natural strain at maximum load. These various data are presented in Figs. 8.7 through 8.13 for the seven steels. In the summary plots, the numerical strength values are presented in detail with the individual curves where each symbol corresponds to one test result; these numerical values will not be further discussed at this time. However, for all steels the tensile and yield strengths drop as the testing temperature is increased from 75 to 350°F. With further increase in testing temperature, the strength values usually indicate 500-degree embrittlement when this phenomenon is characteristic for the steel. This may be and usually is accompanied by a reduction of the yield strength compared to the tensile strength, although there would appear no satisfactory reason to associate these two phenomena. In addition, a steel which has been more heavily damaged by irradiation is the more difficult to postirradiation anneal as determined by these short time tests. Since in some respects radiation damage can be likened to deformation strengthening, these trends are somewhat unexpected.

The reduction of area data for the steels fall in two different groups which have been described as insensitive and sensitive (3). For A212-B, A302-B, and T-1 steels for the neutron fluence of 9.5×10^{19} n/cm^2 >1 Mev, the reduction of area at 75°F is measured as nearly 50 percent. For these steels, the reduction of area tends to be a minimum value when measured at about 550°F, that is, at the temperature of the most pronounced 500-degree embrittlement. The reduction of area values, however, are still in excess of 40 percent, which is an acceptably large value.

For those steels which develop low ductility at 75°F on exposure to a neutron fluence of 9.5×10^{19} n/cm^2 >1 Mev, the ductility remains nearly constant with initial temperature increase followed by an abrupt increase, or it increases gradually with increasing testing temperature until there is a full recovery in ductility at a testing temperature of 650°F as measured by reduction of area. For none of these radiation sensitive steels is there unambiguous evidence of 500-degree embrittlement for reduction of area measurements.

The reduction of area can be used to establish a ductile-brittle transition, and, if this is done, an extremely wide range of brittleness behavior is observed for the steel conditions which have been studied. The ductile-brittle temperatures established in this way do not correspond to those measured in the more conventional impact test, nor do they agree with those that can be determined from the yield/tensile strength ratio and δ_{ML} (Table 8.7).

The yield/tensile strength ratio is usually taken as an index of the uniform strain propagating characteristics of a structural metal, and in this way should be a comparable index of ductility to the natural strain at maximum load (δ_{ML}). The two sets of measurements for the steels are presented in sections (c) and (d) of the respective figures. The yield/tensile strength ratio for convenience to develop a ductile-brittle transition curve of the usual form has been plotted as $(1 - \sigma_{YS}/\sigma_{TS})$. The transition temperatures that have been taken from the several curves are presented in Table 8.7 for comparison with the transition temperatures measured in other ways. It is again emphasized that the transition temperatures taken from the yield/tensile strength ratio and δ_{ML} do not agree with those determined by the reduction of area measurements. On the other hand, these transition temperatures are in reasonable agreement with one another, as they should be, although they both are described in general at higher temperatures than the ductile-brittle transition temperature determined from the impact test. This is a somewhat unusual circumstance in that features of the tensile test results give a more conservative estimate of the ductile-brittle behavior of the steels than is given by the conventional impact test.

Investigations of stress-strain responses of various reactor structural steels are continuing.

Aging Embrittlement of Pressure Vessel Steels

Studies have recently been initiated to investigate another source of embrittlement of pressure vessel steels, that of aging embrittlement. The possible effects of this phenomenon were highlighted by a recent brittle fracture of the Cockenzie boiler drum. The Ducol W.30 steel vessel (5-9/16-in. wall thickness) failed during a preservice hydrotest at a pressure somewhat less than that withstood during several previous hydrotests. This failure is attributed by some to aging embrittlement at the tip of an existing flaw; this resulted in a dynamic situation in that the crack jumped across this embrittled region and propagated at nominal stresses lower than the vessel had previously withstood. It was considered advisable to determine whether or not this effect must be taken into account in the operation of pressurized water reactor (PWR) and boiling water reactor (BWR) pressure vessels that are largely fabricated from A302-B steel.

Aging embrittlement is thought to be of a localized nature affecting the material bordering an existing flaw that is plastically strained during proof test or operation and then is aged at elevated (operating) temperatures. Such embrittlement can be confirmed through the use of a fracture mechanics test since such a test is sensitive to conditions at the crack tip. However, the use of a fracture mechanics specimen of reasonable size (1-in. thick) requires that the tests be performed below the NDT temperature so that the material constraint at the crack tip is high enough to insure valid test results.

In this investigation, fatigue cracked fracture mechanics specimens were preloaded to simulate the proof test and then aged at a conventional PWR operating temperature (550°F). The 1-in. thick notch bend specimens were cut from a 4-in. thick plate (No. 190875) retained at Battelle-Northwest for use by laboratories participating in the coordinated AEC program. This plate was given a conventional heat treatment for reactor pressure vessel applications but was not from a vacuum degassed heat. The NDT temperature of the plate was found to be -90°F from drop weight specimens cut from the 1/4 and 3/4 thickness locations. However, Charpy-V curves from material on both sides of the 1/4 and 3/4 T planes indicate a gradient in properties through the plate thickness. From these data one may project an NDT for center material at least 50°F higher than that experimentally determined at the 1/4 and 3/4 T locations. A linear variation in yield stress on the order of 10 percent was also noted from one surface of the plate to the other. The 2 percent offset yield stress values were determined from 1/4-in. diameter x 7/16-in. long compression plugs loaded slowly in testing. Each plug was oriented with its axis perpendicular to the specimen fracture direction. Yield stress values as a function of temperature are listed in Table 8.8. Because of the observed variation in properties, three groups of notch bend specimens (type and configuration to be described) were taken from 1/4 T, 3/4 T, and center plate locations and were treated separately. The specimen thickness dimension was parallel to the 4-in. plate thickness direction in all cases.

The three-point bend specimen is shown in Fig. 8.14. The dimensions chosen (1-in. thickness T, 16-in. span S, and 5-in. width W) are the same as those for the 1-in. Drop Weight Tear Test (DWTT) specimen used at NRL. This was done as a matter of convenience in case it is later decided to run DWTT in addition to fracture mechanics tests. The specimen proportions are in general agreement with those recommended by the ASTM Committee E24 on Fracture Testing of Metals.

The specimens were subsequently cut with the notch parallel to the primary rolling direction. The notch consisted of a 1/16-in. thick through-thickness slot sharpened at the bottom with a 60 degree mill cutter. The notches were terminated by fatigue cracks that were 0.125-in. to 0.150-in. long making an overall crack dimension (a) of two inches. The specimens were fatigued at room temperature (RT) as cantilever beams using a tension-tension cycle. This imposed nominal bending stresses at the root of the notch that went from 13 percent to 30 percent of yield (about 8 to 19 ksi$\sqrt{\text{in.}}$ applied K level). Approximately 250,000 to 300,000 cycles were required to grow each fatigue crack.

All specimens were then slowly preloaded in three-point bend to a nominal stress at the base of the notch of 75 to 80 percent yield stress (YS) (about 64 ksi$\sqrt{\text{in.}}$) and held for five minutes. One-half of the group was then aged in an oven at 550°F in air for 500 hours; the other half was left at RT. This procedure was meant to represent the conditions of a nuclear vessel during hydrotest and later operation.

Following the aging treatment, the test specimens, including RT control specimens, were loaded in a bend jig at a 4000 lbs/min (11 ksi $\sqrt{\text{in.}}$/min) loading rate. A fracture mechanics type beam gage was inserted into the notch and its output along with the load output from the testing machine was fed into an X-Y recorder. Different test temperatures from RT to -290°F were obtained using liquid nitrogen applied through four chill blocks clamped on both sides of the specimen straddling the fracture path. The temperature was monitored by means of a thermocouple spot welded to the specimen. (Bench tests had initially shown essentially zero temperature variation in the test zone.)

The results of the tests are summarized in Fig. 8.14. The data are presented in terms of the plane strain stress intensity K_{Ic}. This quantity was computed from the following formula of Srawley and Brown (4) for three-point bend.

$$K_{Ic} = \frac{3}{2} \frac{PS\sqrt{a}\,Y}{BW^2} \tag{1}$$

where:

Y = function of $\left(\frac{a}{w}\right)$ as given in the reference

a = crack length

B = thickness

W = width

S = span between supports

P = load

Since all the dimensions (including the crack depth) of all the specimens were very close, the K_{Ic} value was essentially proportioned to the load at fracture instability. All of the load-deflection records with the exception of the upper three points at -90°F were linear, and the instability load is therefore also the maximum value. The foregoing three points do not represent valid fracture mechanics tests because of yielding but are plotted for information using the maximum load in equation (1). Thus, essentially the same pattern of test points would be obtained in a plot of maximum load versus temperature.

Some comments are in order concerning the validity of the results as K_{Ic} values. The fact that all of the load-deflection records were linear up to fracture (with the exception of the three points noted) and that the nominal stress at the root of the notch at fracture was less than 0.9 yield stress are not sufficient criteria for valid results. In addition, the latest recommended practice from ASTM Committee E24 requires that both "a" and "B" be less than

$$2.4\left(\frac{K_{Ic}}{\sigma_{YS}}\right)^2 .$$

These conditions are satisfied by all points at -250°F for both aged and unaged specimens. All of the remaining points satisfy the condition on "a" but not on "B". Wessel (5) has suggested that a value of

$$1.5\left(\frac{K_{Ic}}{\sigma_{YS}}\right)^2$$

for B may be sufficient for A302-B. If this is used, the aged points at -150°F are valid while the unaged ones are less than 20 percent smaller than the thickness requirement. Also, the single 1/4 T material aged point at -90°F is 30 percent less than the thickness requirement.

As seen in Fig. 8.14, there is a trend for the unaged specimens to exhibit a higher fracture toughness than the aged ones for each plate location at temperatures far below NDT. Near the NDT temperature, the effect appears to diminish. At -250°F the average K_{Ic} of three aged specimens from the 3/4 T location shows a K_{Ic} 23 percent below the average of three unaged specimens. For tests from the center plate location at the same temperature, the aged specimens exhibit an average K_{Ic} 15 percent below the average unaged value. At -150°F the aged 1/4 T results are 8 percent below the averaged unaged fracture toughness. Considerable scatter is noted at NDT and no conclusions can be drawn. The upper three points at -90°F are not valid tests and should fall lower if plane strain conditions had been obtained.

Another point to be noted from the figure concerns the relationship of the fractures to the preload level. Some failures for both aged and unaged specimens fell below the preload level at -250°F while at -150°F and higher they all fell above this level. To check the behavior exhibited at -250°F, one unaged specimen from the 3/4 T location that exhibited failures <u>above</u> the preload level at -250°F was preloaded again but at this time to 93 percent of RT yield stress. This would seem to indicate that the failure of even unaged specimens can occur below the preload level and therefore has implications for the use of warm prestressing in vessel operation. In general, the results show a pattern to be expected for specimens without preload, namely, a rise in toughness with temperature. However, it is not unexpected that failures would occur at levels higher than the preload level even at temperatures below NDT. This would be the case if the applied preload was less than the load required for failure below NDT in a virgin specimen. Evidently, this was the case, but no unpreloaded specimens were available to provide a check.

These preliminary results point out that there may be an effect of aging embrittlement on this plate of A302-B steel, and the phenomenon should be further investigated. Additional tests are required before general conclusions can be drawn. However, one implication concerning vessel operation below NDT can be made. If a vessel is proof tested below NDT to 1.25 times its design pressure and a flaw exists that is just subcritical, then the scatterband of data at -250°F indicates that the aging embrittlement effect could cause failure upon subsequent pressurization at the same temperature at 85 percent of design pressure. This approaches the operating level of a nuclear vessel. It is planned to continue this investigation using another heat of A302-B steel and other steels.

Post-Pressurization Test Operations on PM-2A Reactor Prssure Vessel

The Naval Research Laboratory has been assigned the responsibility for coordinating efforts to obtain and test metallurgical specimens from the PM-2A reactor pressure vessel as required by the third, or post-pressurization phase, of planned vessel tests. Through its contract with the AEC, NRL has arranged for the Idaho Nuclear Corporation (INC) to physically remove a steel test section from the vessel, to section it into appropriate specimen blanks, and to finish machine these blanks into tensile, Charpy V-notch, drop weight, and wedge opening loaded (WOL) test specimens, Fig. 8.15.

At the beginning of FY 68, INC shipped the first series of tensile, Charpy-V notch, and drop weight test specimens to NRL for evaluation in the High Level Radiation Laboratory (HLRL). Concurrently, the WOL specimens were passing through rough and final machining stages. These specimens will be shipped to Battelle Memorial Institute (BMI-Columbus) for fatigue cracking (or notching by EDM method as necessary) and for testing.

Following the pressurization phase of the PM-2A vessel tests, the first objective was to remove samples of the vessel steel for chemical analyses and for neutron fluence determinations. The chemical composition subsequently determined by the Chemical Processing Plant at the National Reactor Testing Station was in excellent agreement with the nominal composition of A350-LF3 steel and with that given by the fabrication mill test reports. The neutron fluence values ranged from a maximum of 1×10^{19} n/cm^2 >1 Mev at the fracture itself (at vessel/clad interface) to a low of about 1.6×10^{18} n/cm^2 >1 Mev at a location on the outside diameter of the vessel at a point about two feet circumferentially around the vessel from the top of the artificial flaw.

The results of specimen tests as well as plans for the removal of additional steel for further testing will be presented in future reports.

Gage Marking Device for Irradiated Tensile Specimens

Significant problems have occurred as a result of employing a conventional indentor to establish an initial 1-in. reference gage length on radioactive tensile specimens. In many cases the loss of ductility following irradiation is so severe that the indentations are a sufficient enough stress raiser to localize fracture. When this occurs, valuable data are lost since there is no reference from which the amount of elongation can be determined. In addition, the question of possible premature specimen failure is always raised in such cases.

The first attempt to eliminate this problem was to paint the reduced section of the tensile specimen; then the 1-in. gage reference lines were scribed in the paint using a soft aluminum scriber. This proved to be unsatisfactory for elevated temperature testing above 500°F since the paint would not adhere to the smooth surface of the specimen.

An alternate approach which uses an electrolytic etching technique was then tried and proved to be very effective. A small portable etcher is installed in a fixture and hinged to a stand as shown in Fig. 8.16. A spring holds the etcher in the "ready" position while V-blocks permit accurate positioning of the specimen. The 1-in. reference gage length is provided by placing a 1-in. strip of teflon across the felt pad containing the electrolyte. Then, when the etcher is pressed down against the specimen, and current is passed through the unit, two etched areas spaced precisely one inch apart are produced. Post-test elongation measurements are made by using a special holding and gaging fixture (6).

This etcher has now been placed in routine operation in the tension testing cell. A distinguishing feature of this unit is that the marking capability is not limited to one inch as is the conventional indentor, and further, various sized specimens can be accommodated with only minor changes.

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(5) E. T. Wessel, "State of the Art of the WOL Specimen for K_{Ic} Fracture Toughness Testing," Westinghouse Research Laboratories, Research Report 67-1D6-BTLER-R1, March 22, 1967.

(6) L. E. Steele, et al, "Irradiated Materials Evaluation and Reactor Pressure Vessel Surveillance for the Army Nuclear Power Program," NRL Memorandum Report 1644, September 1, 1965.

TABLE 8.1

Chemical Composition and Heat Treatment of A533 Grade B, Class I Steel From a Vacuum Degassed Electric Furnace Heat

Material Form	Chemical Analysis (Wt %)									Heat Treat-ments
	C	Mn	P	S	Si	Ni	Mo	Cu	Al	
4-in.Plate	.21	1.40	.010	--	.25	.50	.45	.15	.048	(1)
8-in.Plate	.19	1.37	.012	.011	.25	.52	.45	--	--	(2)

Heat Treatments:

(1) Austenitized at 1650°F for 4 hours, water dip quenched; tempered at 1250°F for 10 hours, furnace cooled; stress relief annealed at 1150°F for 20 hours, furnace cooled.

(2) Austenitized at 1650°F for 8 hours, water dip quenched; tempered at 1250°F for 10 hours, furnace cooled; re-austenitized at 1550°F for 8 hours, water spray quenched; tempered at 1260°F for 8 hours, air cooled; stress relief annealed at 1150°F for 20 hours, furnace cooled.

TABLE 8.2

Chemical Composition and Heat Treatment of

3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V Test Plates Irradiated at <250°F

Steel	Thickness (in.)	Chemical Analysis (%)											Heat Treatments
		C	Mn	Si	P	S	Ni	Cr	Mo	V	Cu	Al	
3-1/2Ni-Cr-Mo	8	.17	.38	.29	.013	.023	3.65	1.88	.51	-	-	.02	(1)
5Ni-Cr-Mo-V	1	.12	.74	.22	.008	.005	5.06	.63	.50	.07	.06	-	(2)

Heat Treatments:

(1) Austenitized at 1650°F with 8 hours heating and 2 hours hold, water quenched 17 minutes; re-austenitized at 1500°F with 8 hours heating and 2 hours hold, water quenched 17 minutes; tempered at 1185°F with 8 hours heating and 2 hours hold, water quenched cold.

(2) Standard mill quench and tempering treatment for 130 ksi minimum yield strength.

TABLE 8.3

Charpy-V Notch Ductility Performances of Irradiated 3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V Steel Plate

Both Parallel and Transverse to Their Primary Rolling Direction

A. 8-in. 3-1/2Ni-Cr-Mo Plate (1.6 × 10^{19} n/cm^2 >1 Mev at <250°F)

Orientation to Rolling Direction	Irradiation Condition: C_V 30 ft-lb Transition Temp. (°F)			Irradiation Condition: Full Shear Energy Absorption (ft-lb)			Postirradiation Annealed Condition (168 hr) (C_V 30 ft-lb Transition Temp. (°F))				
	Initial	Irradiated	ΔT	Initial	Irradiated	Loss	Irradiated	Annealed 650°F	ΔT Recovery	Annealed* 750°F	ΔT Recovery
Parallel	-130	120	250	73	54	19	120	-95	215	-130	250
Transverse	- 95	165	260	59	41	18	165	-60	225	- 95	260

*Full recovery in energy absorption indicated.

B. 1-in. 5Ni-Cr-Mo-V Plate (5.5 × 10^{18} n/cm^2 >1 Mev at <250°F)

Orientation to Rolling Direction	Initial	Irradiated	ΔT	Initial	Irradiated	Loss	Postirradiation Annealed Condition
Parallel	-250	-100	150	110	81	29	
Transverse	-215	- 70	145	91	64	27	

TABLE 8.4

Chemical Composition and Heat Treatment of A353 Steel

used in the Retained Austenite Study

NRL Code	Plate Thickness (in.)	Chemical Analysis (%)												Retained Austenite (%) (x-ray Diffraction)
		C	Mn	P	S	Si	Ni	Cr	Mo	V	Al	Cu	Ti	
R-1[a] *	1	.11	.42	.008	.012	.25	8.90	.07	.02	-	-	.07	.02	3.3
R-2[b] *	1	.11	.42	.008	.012	.25	8.90	.07	.02	-	-	.07	.02	7.2
R-3[c] *	1	.11	.42	.008	.012	.25	8.90	.07	.02	-	-	.07	.02	7.1
R-4[d] *	1	.11	.42	.008	.012	.25	8.90	.07	.02	-	-	.07	.02	11.2
IN[e] *	1	.09	.50	.012	.025	.27	8.99	-	-	-	-	-	-	-
IN[e]	1	.10	.47	.008	.023	.29	8.99	.06	.15	-	-	-	-	-
F-53[f]	2	.09	.40	.009	.017	.21	8.50	.10	.03	.02	.01	-	-	-

[a] Mill quenched and tempered plate reheated to 1475°F, water quenched, tempered at 1050°F for 10 hours, air cooled.

[b] Mill quenched and tempered plate reheated to 1475°F, water quenched, tempered at 1050°F for 60 hours, air cooled.

[c] Mill quenched and tempered plate reheated to 1475°F, water quenched, tempered at 1100°F for 7 hours, air cooled.

[d] Mill quenched and tempered plate reheated to 1475°F, water quenched, tempered at 1050°F for 811 hours, air cooled, re-tempered at 1075/1085°F for 479 hours, air cooled.

[e] Air cooled from 1650°F, re-heated to 1450°F and air cooled, tempered at 1050°F. air cooled.

[f] Water quenched from 1475°F, tempered 2 hours at 1100°F, water quenched.

*Analysis by supplier.

TABLE 8.5

Charpy-V Transition Temperature Behavior of A353 Steel

with Varying Amounts of Retained Austenite after Irradiation

at $<250°F$ to 7.3×10^{19} n/cm^2 >1 Mev

NRL Code	Retained Austenite (%)	Charpy-V 30 ft-lb Transition Temperature (°F)			Full Shear Energy Absorption (ft-lbs)	
		Initial	Irradiated	ΔT	Initial	Irradiated
R-1	3.3	-345	95	440	83	36
R-2	7.2	-355	20	375	99	48
R-3	7.1	-335	55	390	86	43
R-4	11.2	-410	-10	400	90	40
IN	--	-300	210	510	73	44
F-53	--	-340	95	435	87	39

TABLE 8.6

Tensile Properties of A353 Steel with Varying Amounts

of Retained Austenite Before and After Irradiation

at $< 250°F$ to 6.9×10^{19} n/cm^2 $>$1 Mev

NRL Code	Retained Austenite (%)	Yield Strength (ksi)			Tensile Strength (ksi)			Elongation (%)		Reduction of Area (%)	
		Initial	Irrad.	Ave. Δ	Initial	Irrad.	Ave. Δ	Initial	Irrad.	Initial	Irrad.
R-1	3.3	101.0	159.8	58.0	110.0	160.2	49.3	27.5	9.3*	71.7	59.2
		100.5	157.6		109.5	157.8		25.4	7.6*	69.6	52.3
R-2	7.2	94.0	154.2	60.4	105.5	154.2	49.3	28.2	11.3	70.0	52.9
		93.2	153.8		104.0	153.8		29.3	*	74.2	47.3
R-3	7.1	90.8	146.6	56.9	111.2	146.6	36.8	29.2	15.7	68.2	57.1
		88.6	146.6		108.5	146.6		29.4	15.3	65.5	56.6
R-4	11.2	72.2	138.0	63.8	92.2	138.5	44.7	36.4	14.4	74.6	55.0
		75.0	136.8		94.0	137.0		36.8	14.0	71.3	60.2
IN	--	94.2		**	111.0		**	28.6	**	67.9	**
	--	99.7			114.0			27.6		68.9	
	--	92.6			109.5			28.7		67.3	
	--	98.7			114.2			31.7		70.4	
	--	98.3			113.7			29.7		68.7	
	--	93.5			110.0			28.6		67.3	
F-53	--	106.3	155.2	52.2	115.9	155.5	42.8	27.5	10.4*	70.0	58.7
	--	104.5	155.2		114.8	155.8		27.3	13.1	66.4	59.2
	--	98.3			107.0			26.3		70.4	

*Pinprick in reduced area.

**Specimens of this material were not irradiated.

TABLE 8.7

Ductile-Brittle Transition Temperatures

As Determined by Several Experimental Methods

Type of Steel	Neutron Fluence (10^{19} n/cm^2 >1 Mev)	Transition Temperatures (°F)				
		Charpy-V (30 ft-lb)	Drop Weight (NDT)	Reduction of Area (%)	YS/TS	δ_{ML}
A312-B	0	+ 5	- 30	< 75	-	-
	0.85	+250	-	< 75	250	325
	1.1	+255	+230	-	-	-
	2.3	+295	-	< 75	400	375
	9.5	+385	-	< 75	500	450
A302-B	0	+ 15	+ 10	< 75	-	-
	0.85	+245	-	< 75	325	275
	1.8	+315	+300		-	-
	2.3	+315	-	< 75	425	400
	9.5	+415	-	< 75	550	525
T-1	0	-105	-100	< 75	-	-
	0.8	+ 70	-	-	-	-
	2.3	-	-	< 75	450	375
	9.5	-	-	< 75	600	625
A350-LF1	0	- 45	- 40	< 75	-	-
	0.8	210	-	-	-	-
	2.3	285	-	< 75	400	300
	5.3	395	400	-	-	-
	9.5	-	-	~300	550	550
A350-LF3	0	- 80	- 80	< 75	-	-
	0.85	+160	-	< 75	325	325
	2.3	+300	-	< 75	400	375
	9.5	+500	-	~400	>650	>650
HY-80	0	-220	-190	< 75	-	-
	0.85	-	-	< 75	350	250
	2.3	+ 50	-	< 75	450	350
	9.5	+300	-	~550	650	600
A353	0	-295	-	< 75	-	-
	0.8	-150	-	-	-	-
	2.3	-	-	< 75	350	350
	9.5	~ +220	-	~300	450	450

TABLE 8.8

Yield Stress Values for A302-B Plate

Plate Location	Yield Stress (ksi)	Temperature (°F)
1/4 T	74.6	+75 (Room Temperature)
Center	75.5	
3/4 T	81.0	
1/4 T	83.9	
Center	86.5	- 90
3/4 T	92.5	
1/4 T	94.4	
Center	95.0	-150
3/4 T	98.2	
1/4 T	122.7	
Center	118.0	-250
3/4 T	124.9	

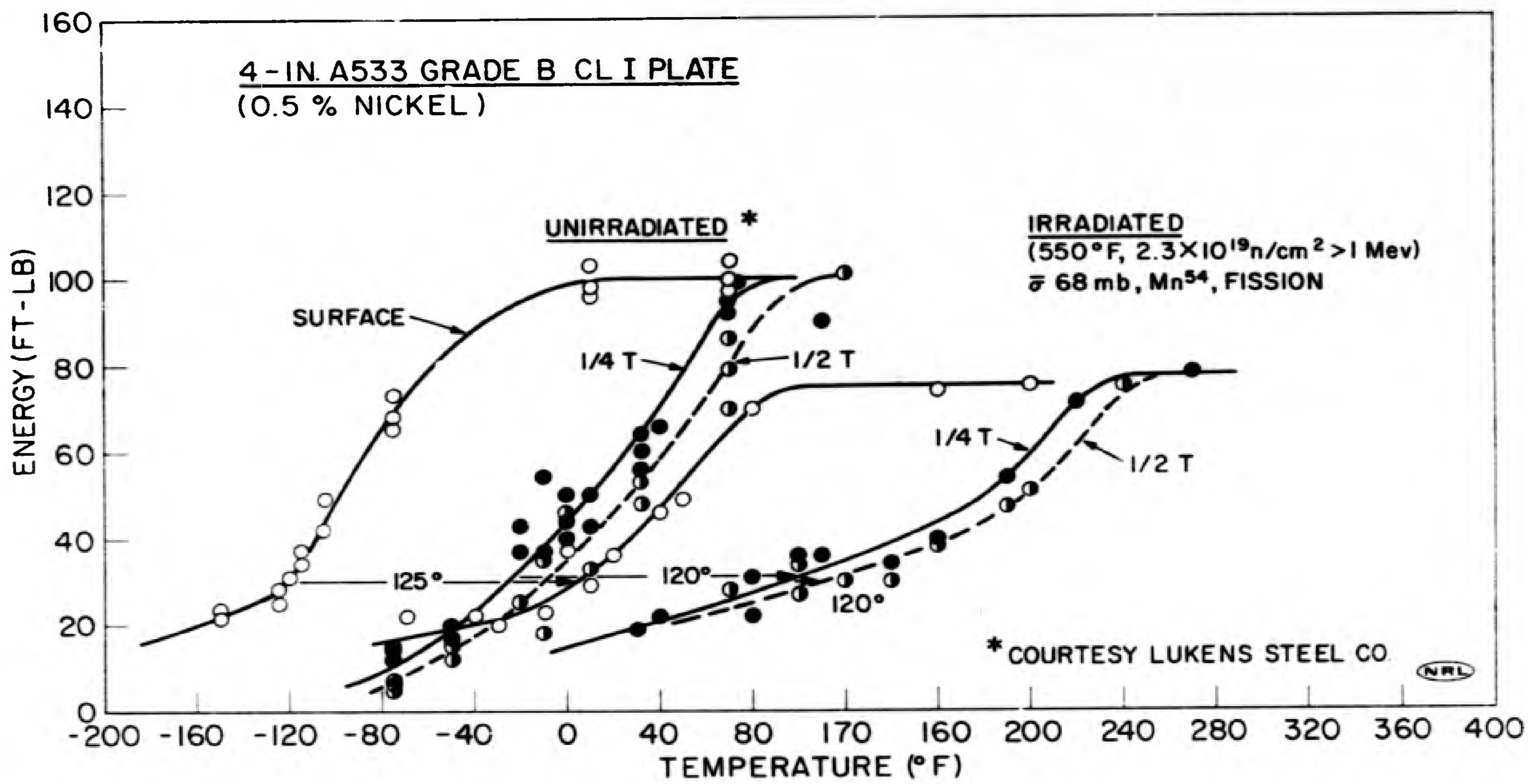

Fig. 8.1 - Charpy-V notch ductility behavior of 4-in. A533-Grade B, Class I steel showing through-thickness response to neutron irradiation at 550°F

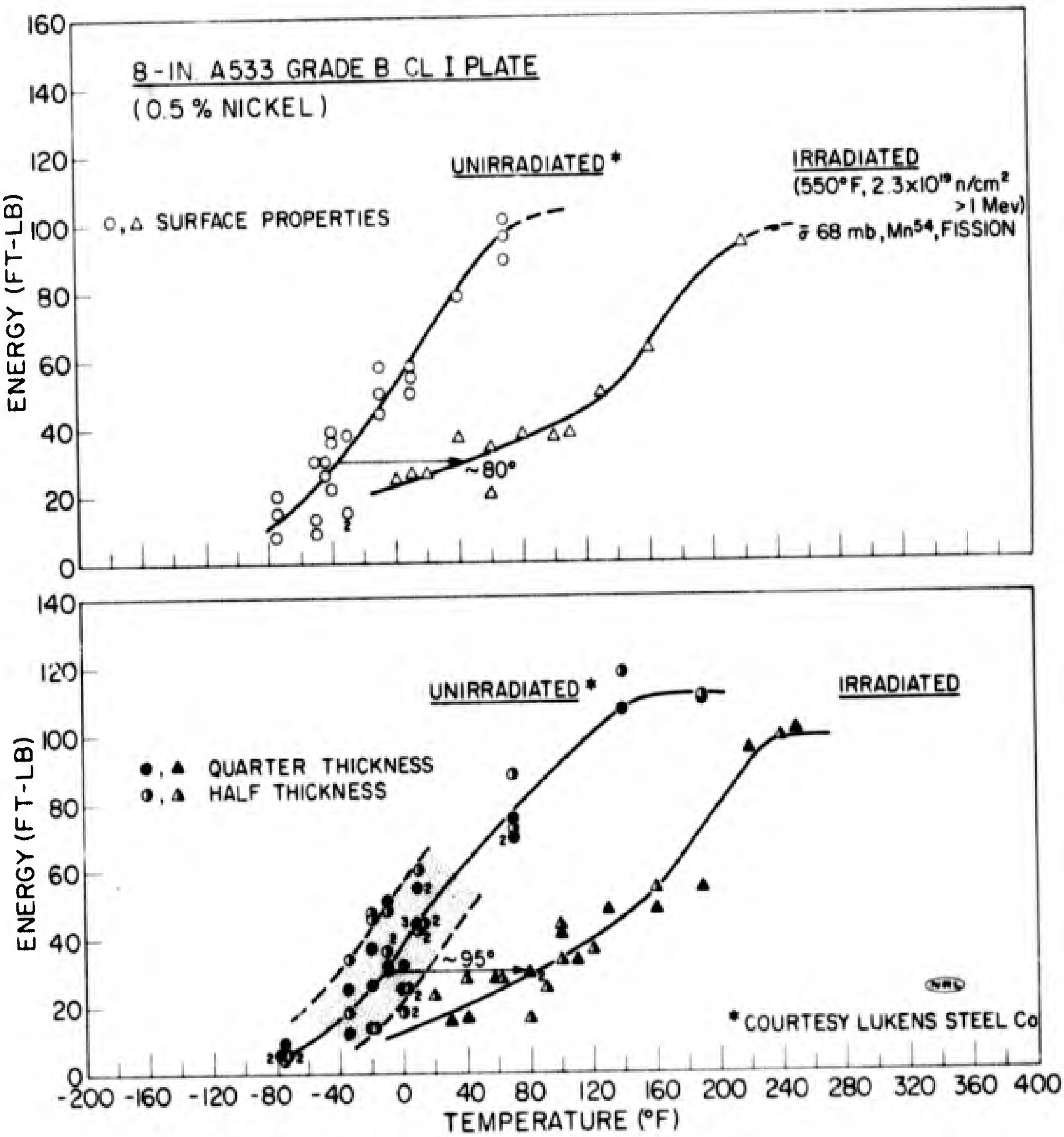

Fig. 8.2 - Charpy-V notch ductility behavior of 8-in. A533-Grade B, Class I steel showing through-thickness response to neutron irradiation at 550°F. Pre- and postirradiation properties of the plate surface layer have been plotted separately for visibility.

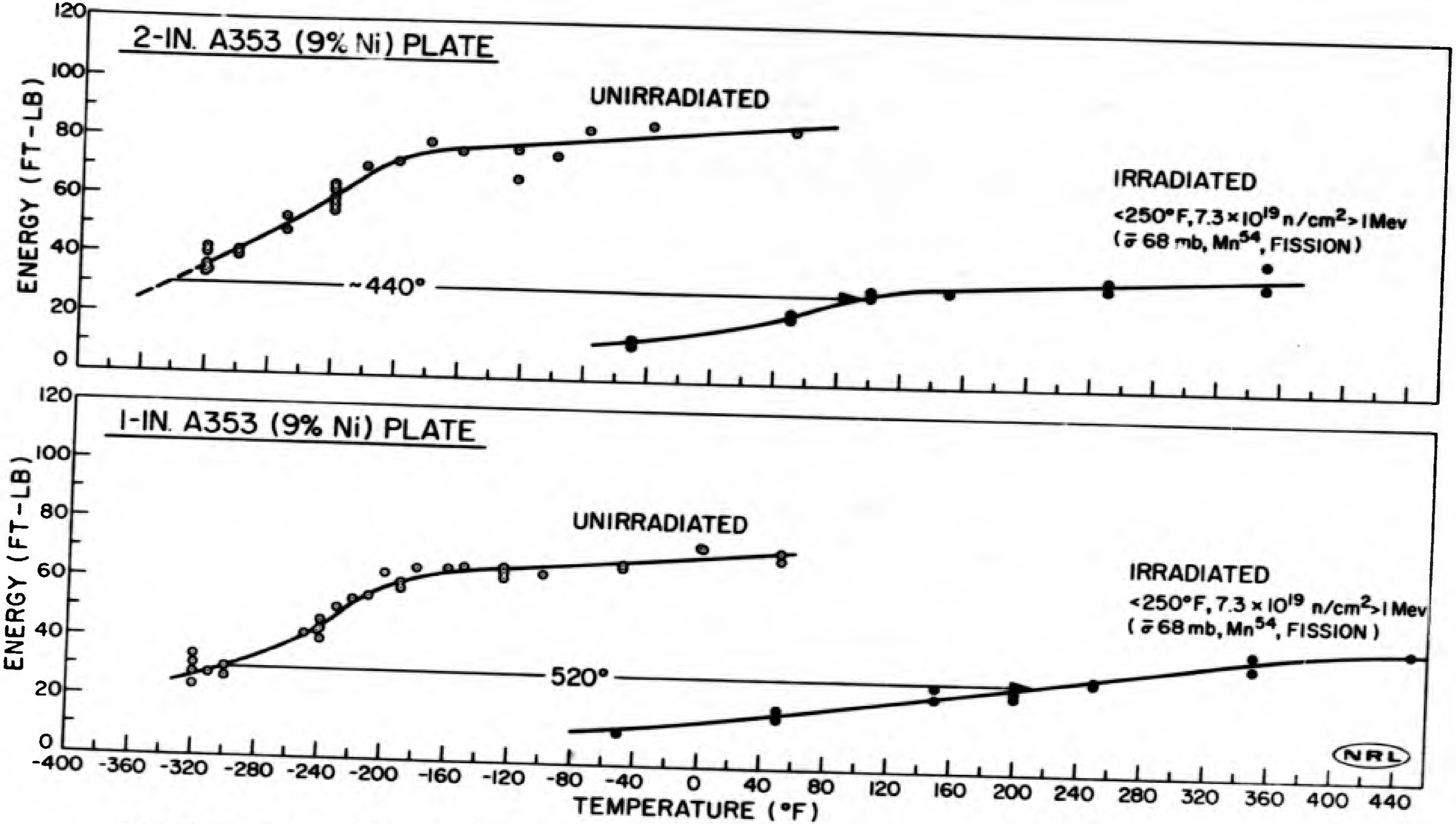

Fig. 8.3 - Charpy-V notch ductility characteristics of 2-in. quenched and tempered and 1-in. double normalized and tempered A353 plate before and after exposure to a high neutron fluence, (7.3×10^{19} n/cm^2 >1 Mev) at a temperature of <250°F

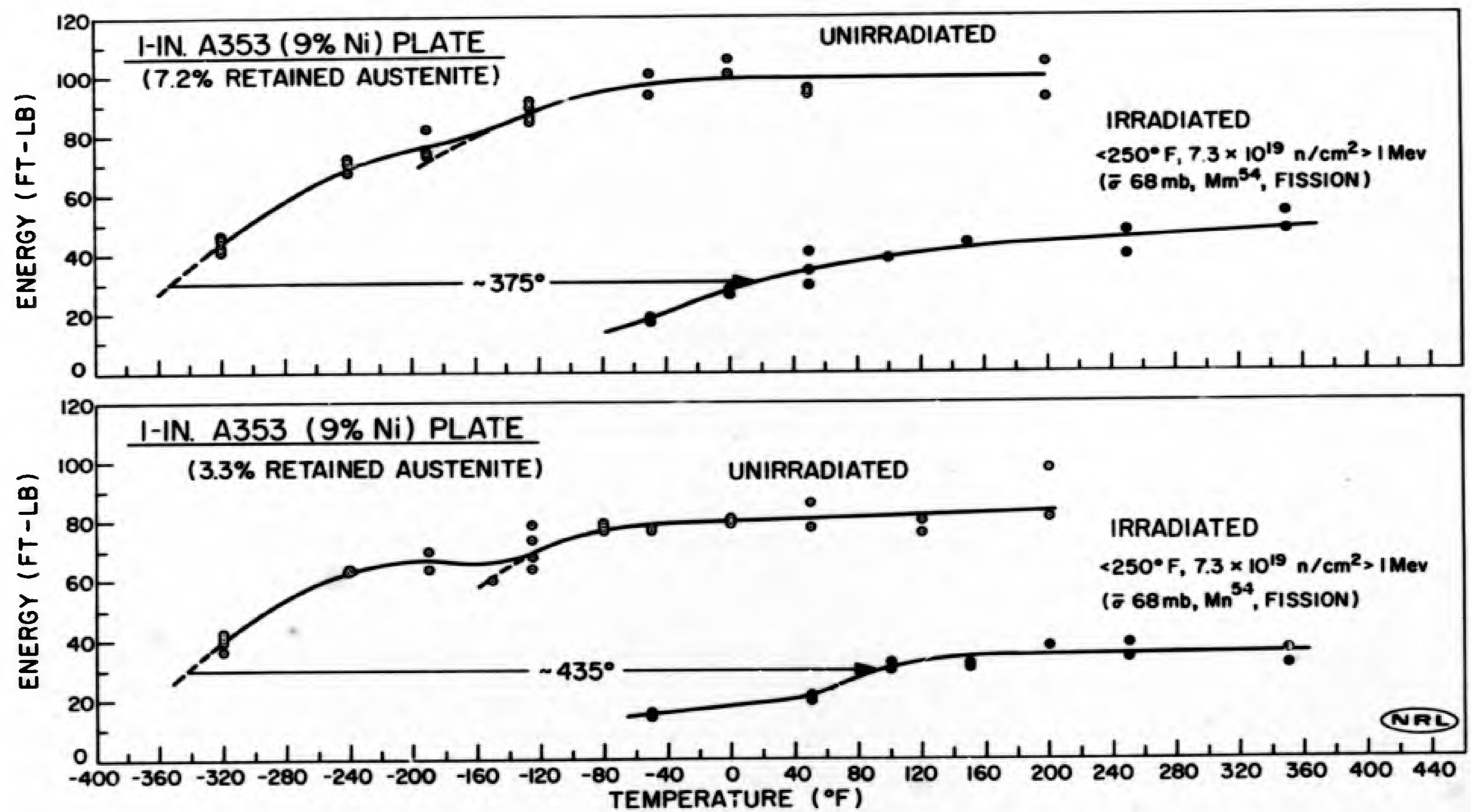

Fig. 8.4 - Charpy-V notch ductility characteristics of experimentally heat treated 1-in. A353 plates containing 3.3% and 7.2% retained austenite before and after exposure to a high neutron fluence, (7.3×10^{19} n/cm ^{2} >1 Mev) at a temperature of >250°F

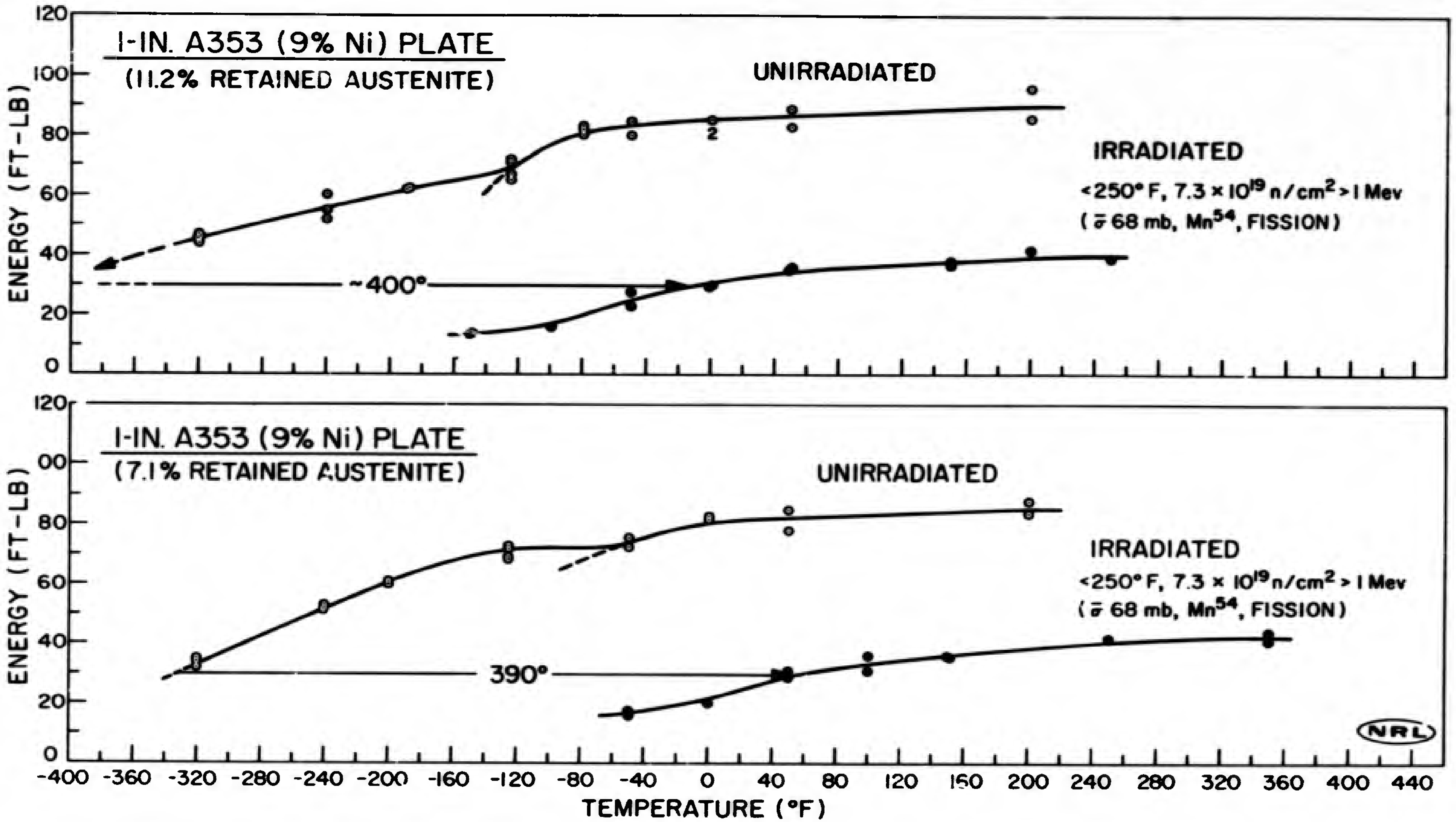

Fig. 8.5 - Charpy-V notch ductility characteristics of experimentally heat treated 1-in. A353 plates containing 7.1% and 11.2% retained austenite before and after exposure to a high neutron fluence, (7.3×10^{19} n/cm^2 >1 Mev) at a temperature of <250°F

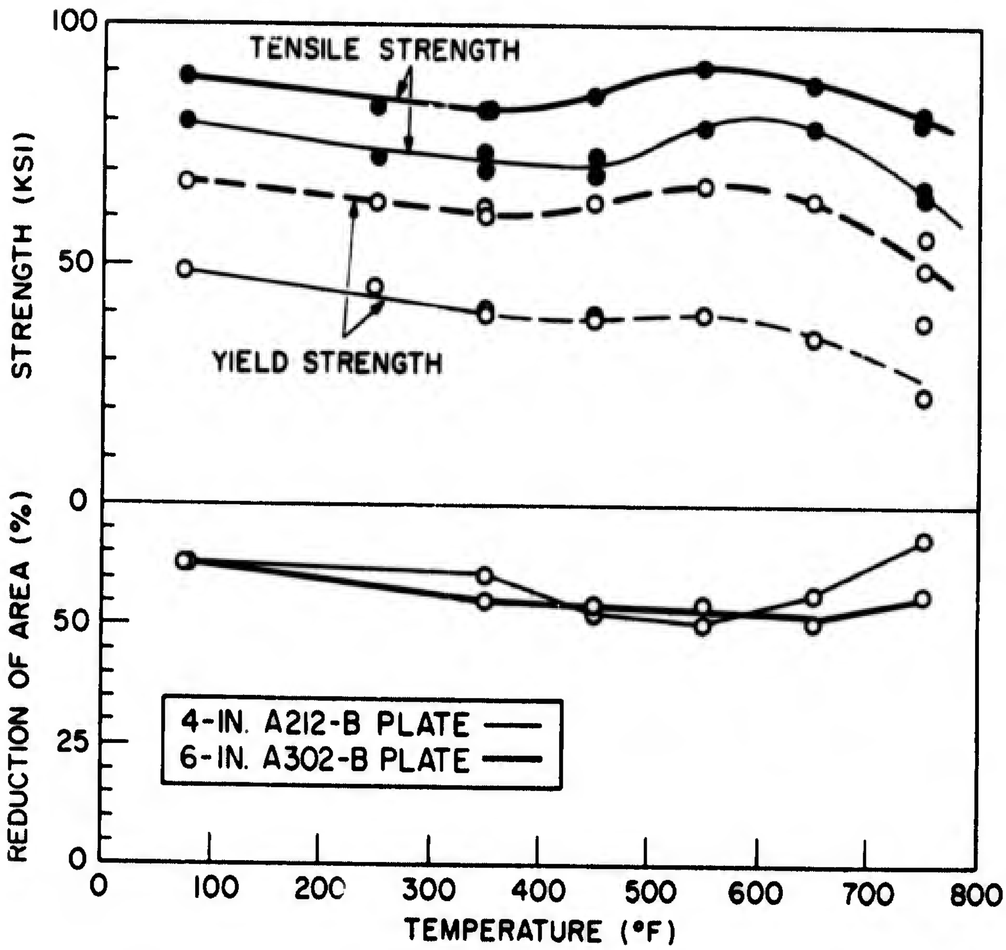

Fig. 8.6 - The tensile properties of unirradiated A212-B and A302-B steels to 750°F

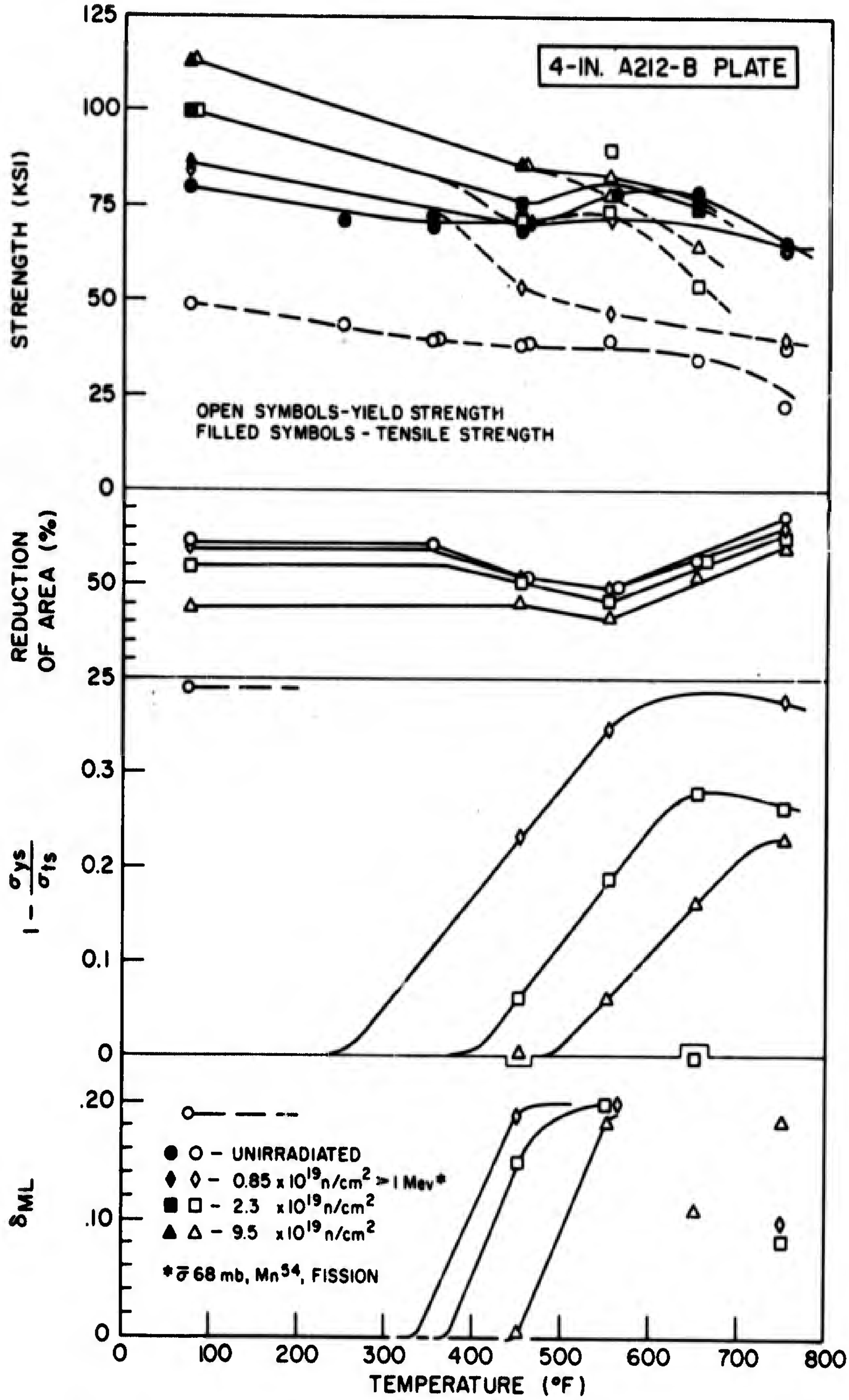

Fig. 8.7 - The tensile properties of A212-B to 750°F for the indicated irradiation conditions

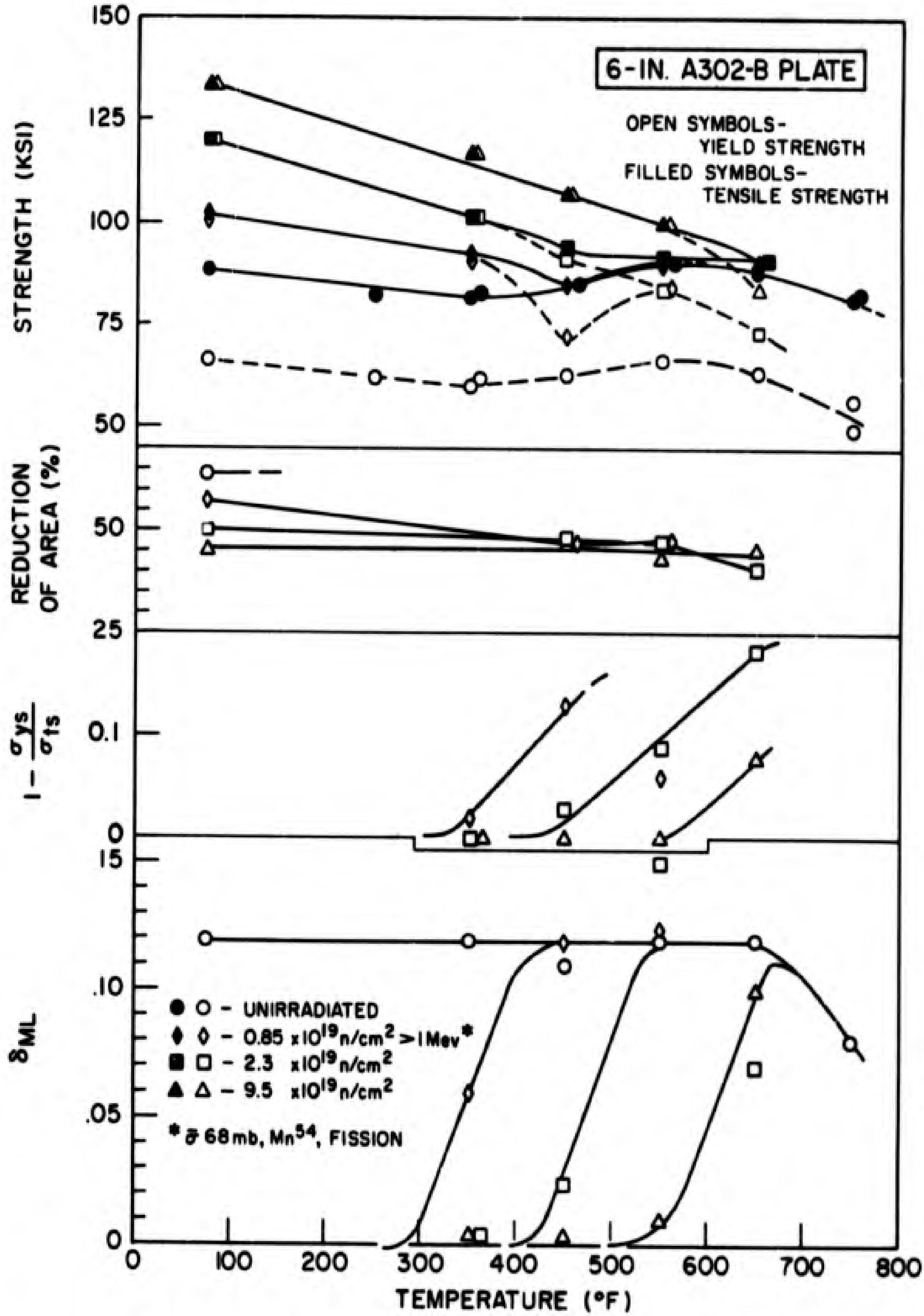

Fig. 8.8 - The tensile properties of A302-B steel to 750°F for the indicated irradiation conditions

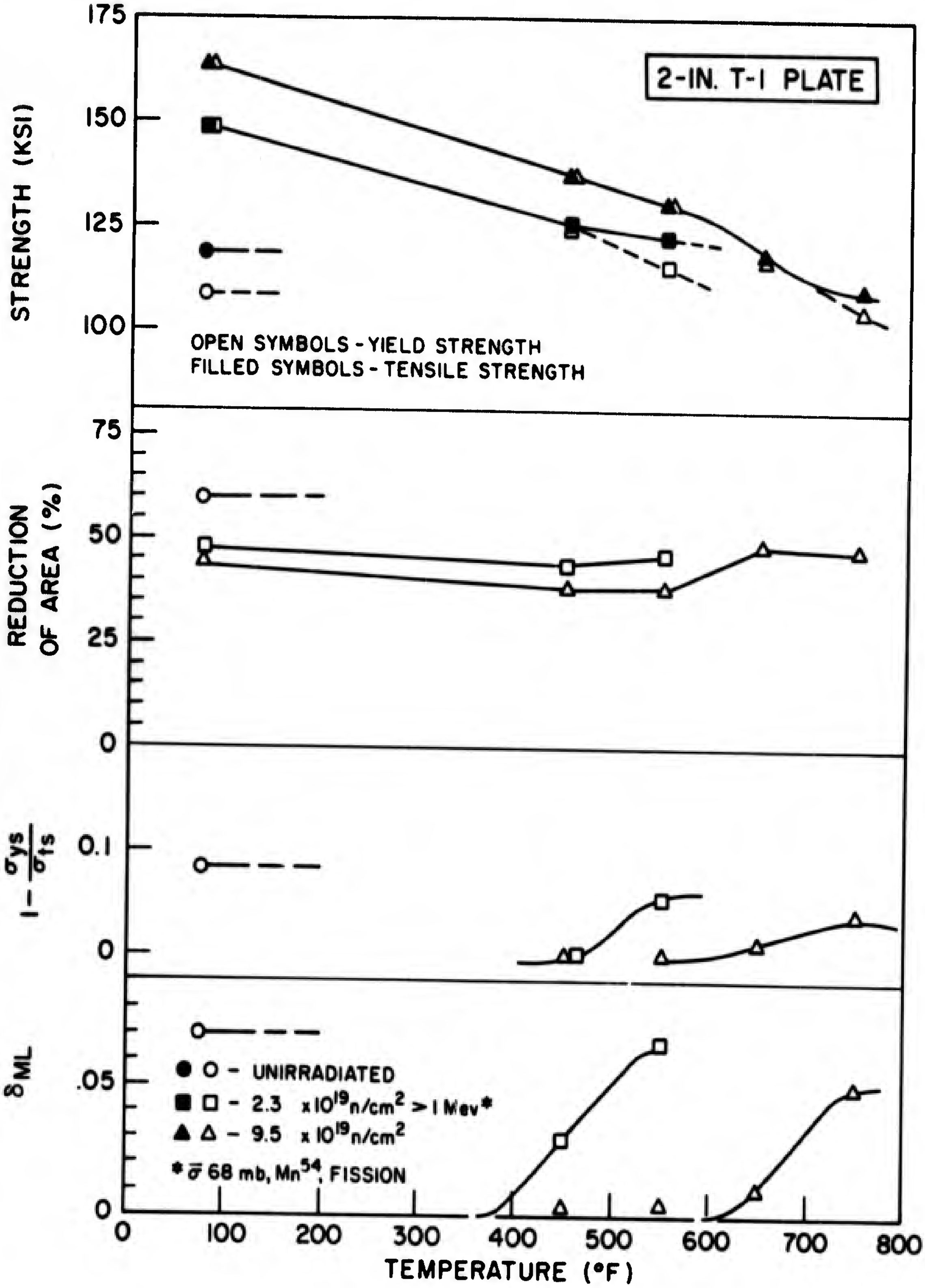

Fig. 8.9 - The tensile properties of T-1 steel to 750°F for the indicated irradiation conditions

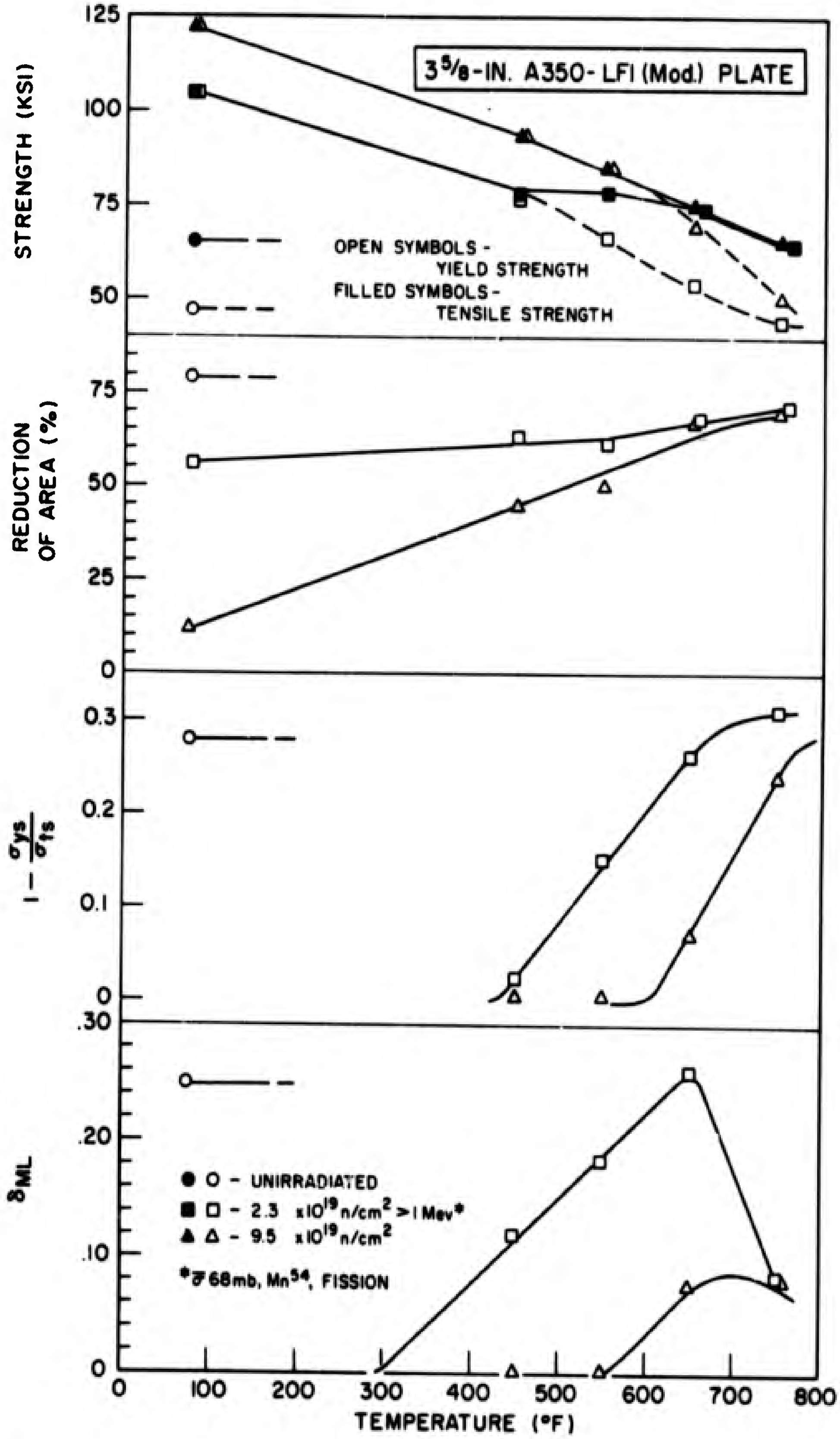

Fig. 8.10 - The tensile properties of A350-LF1 (Mod.) steel to 750°F for the indicated irradiation conditions

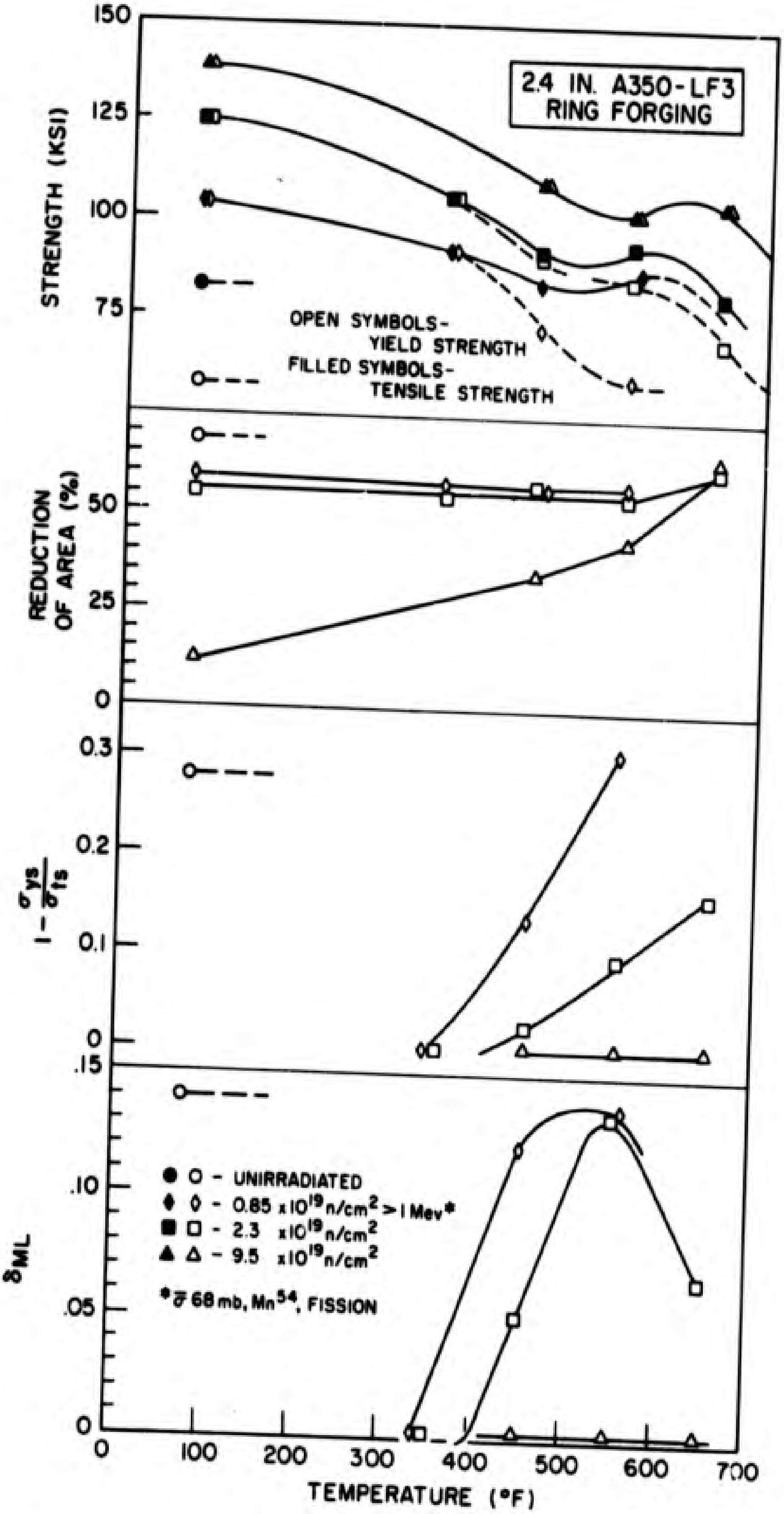

Fig. 8.11 - The tensile properties of A350-LF3 steel to 750°F for the indicated irradiation conditions

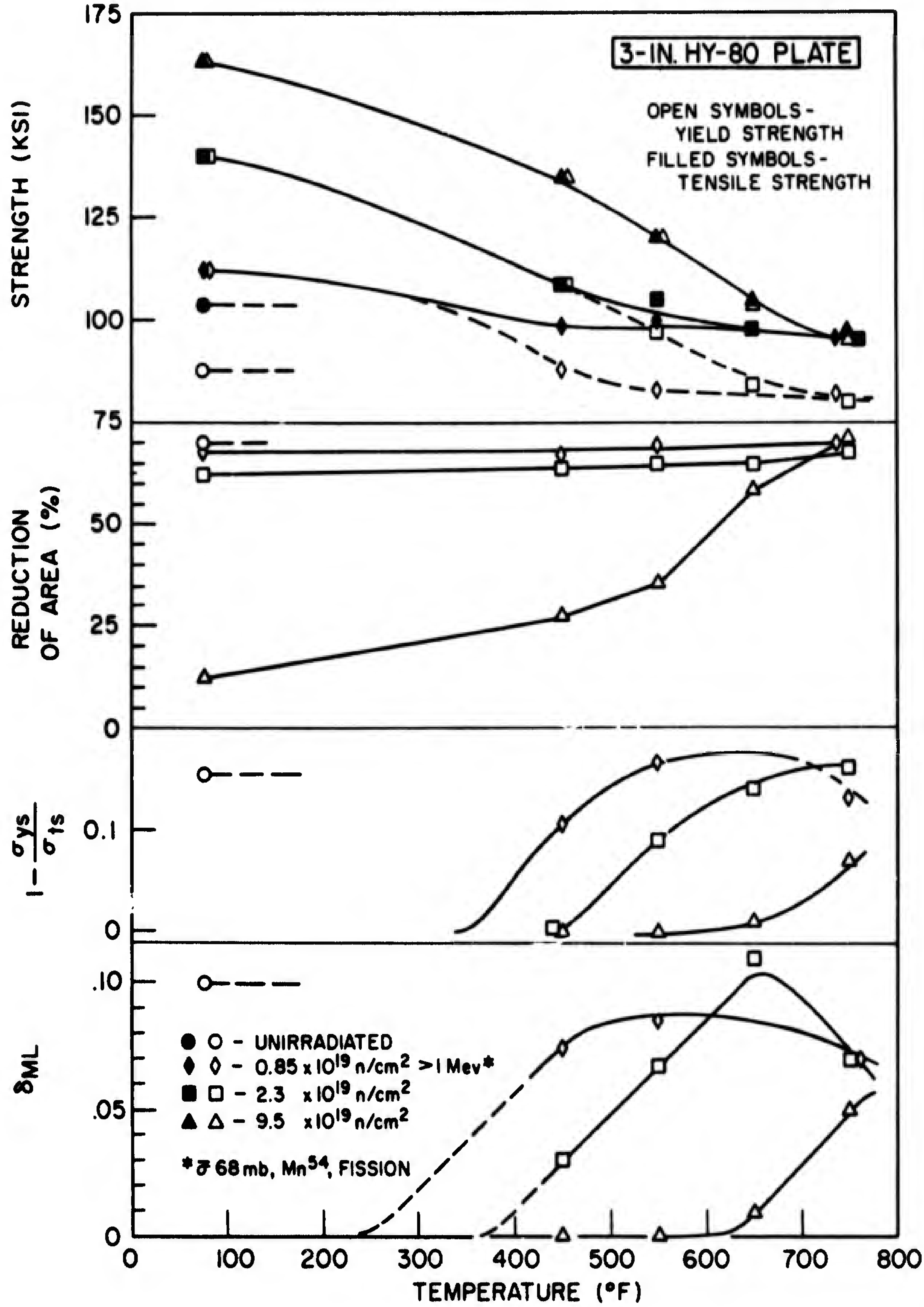

Fig. 8.12 - The tensile properties of HY-80 steel to 750°F for the indicated irradiation conditions

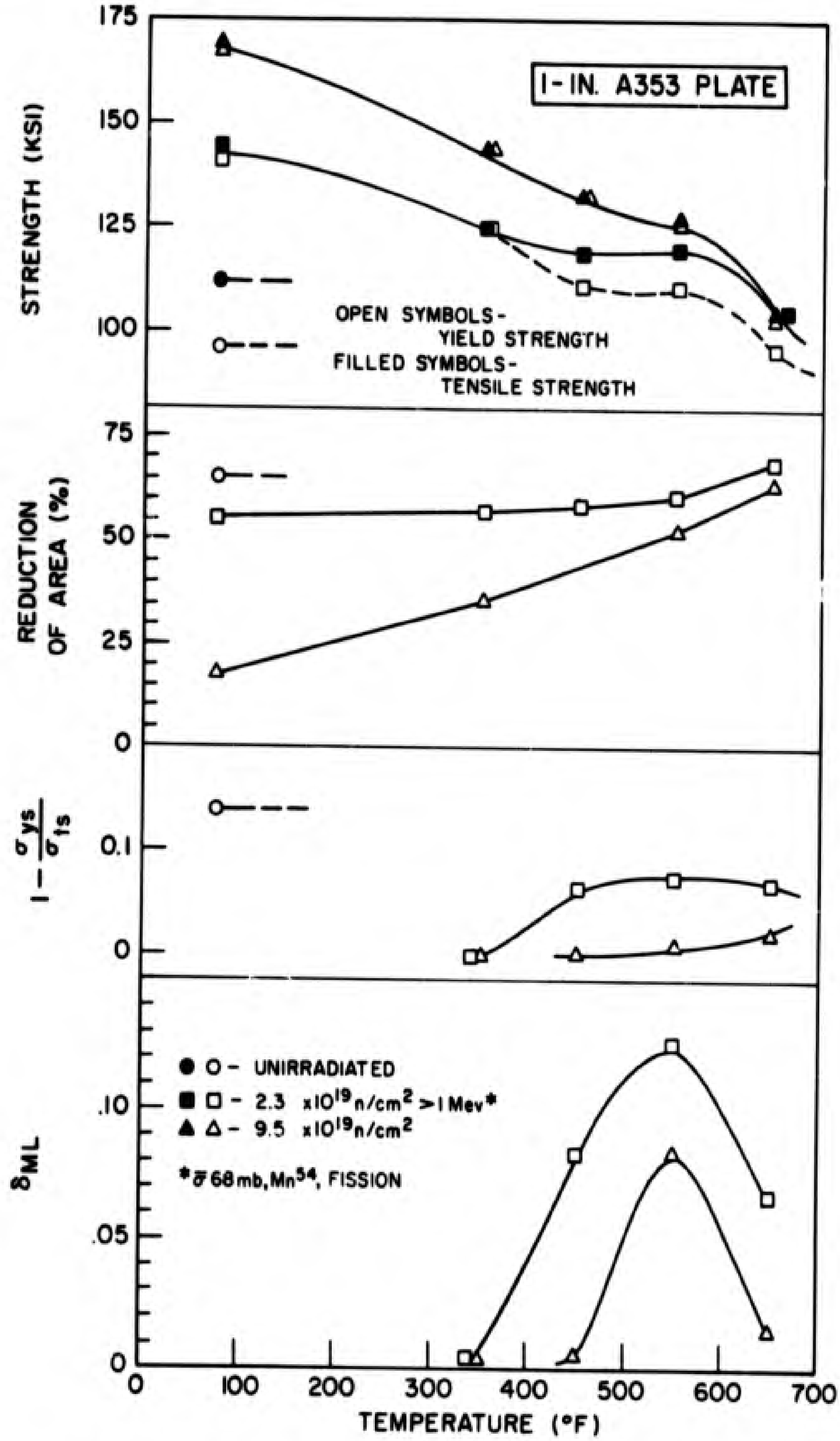

Fig. 8.13 - The tensile properties of A353 steel to 750°F for the indicated irradiation conditions

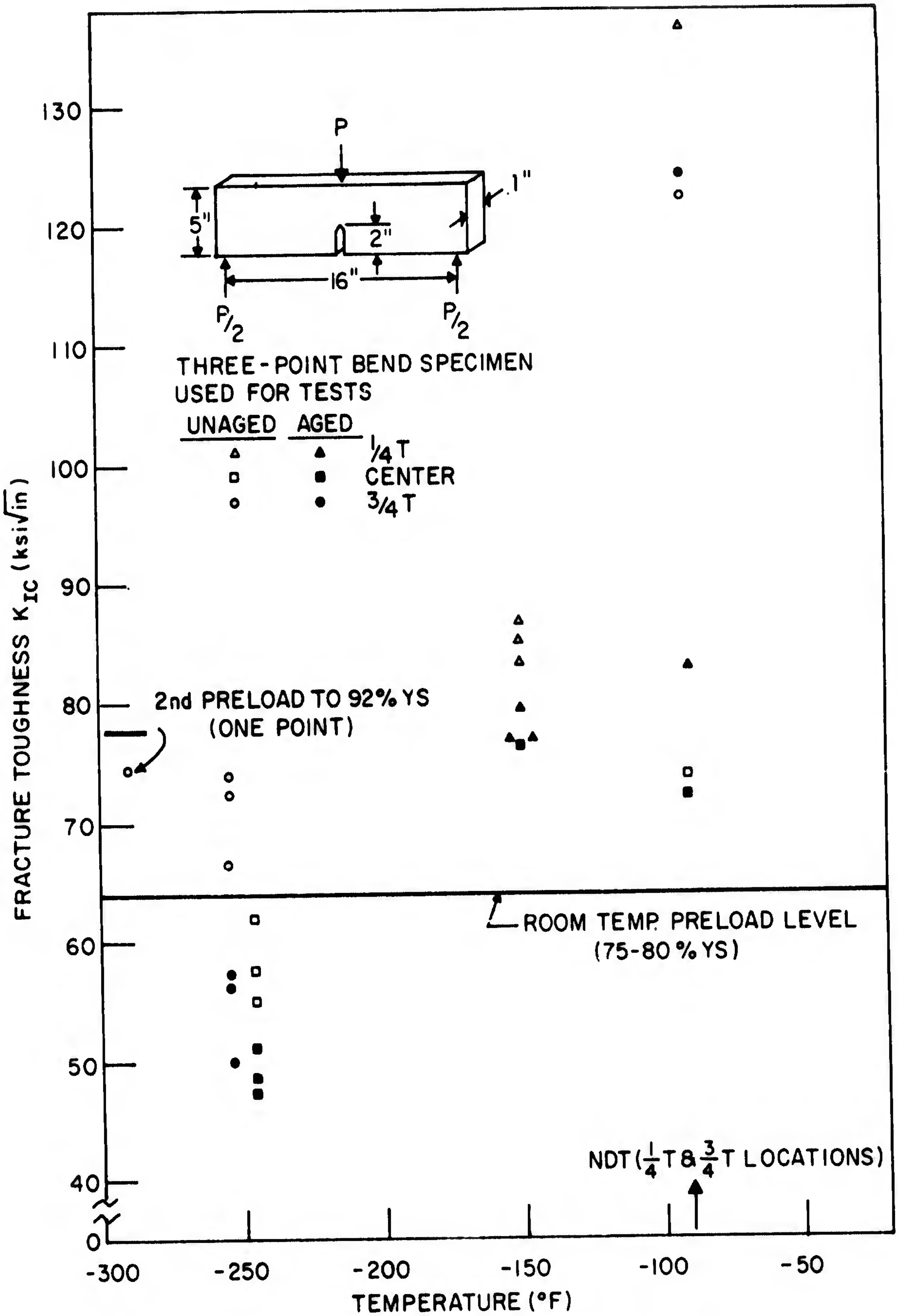

Fig. 8.14 - Fracture toughness of aged and unaged A302-B steel specimens at various temperatures

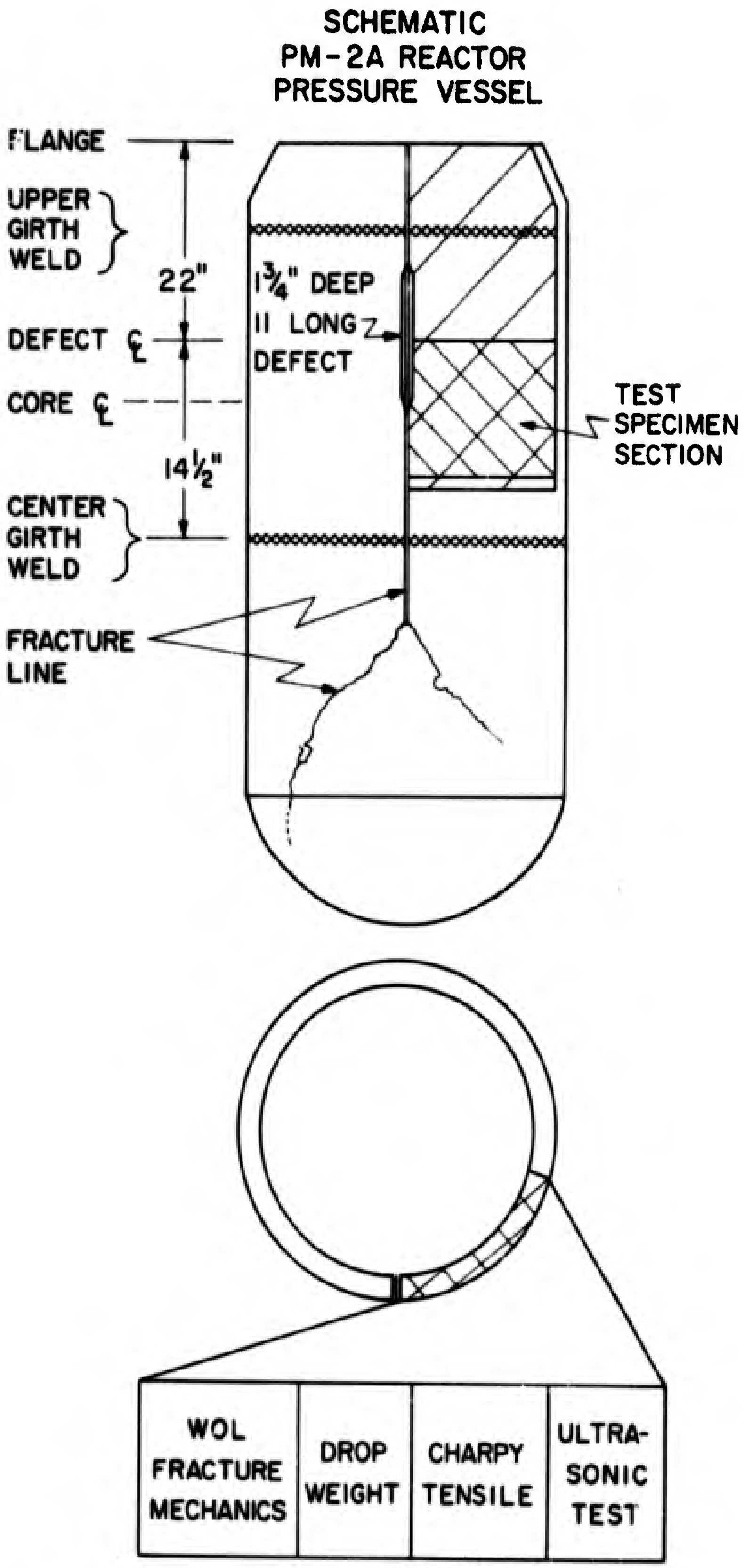

Fig. 8.15 - Schematic drawing of the PM-2A reactor pressure vessel showing the artificial defect, fracture, and location of material removed for metallurgical test specimens

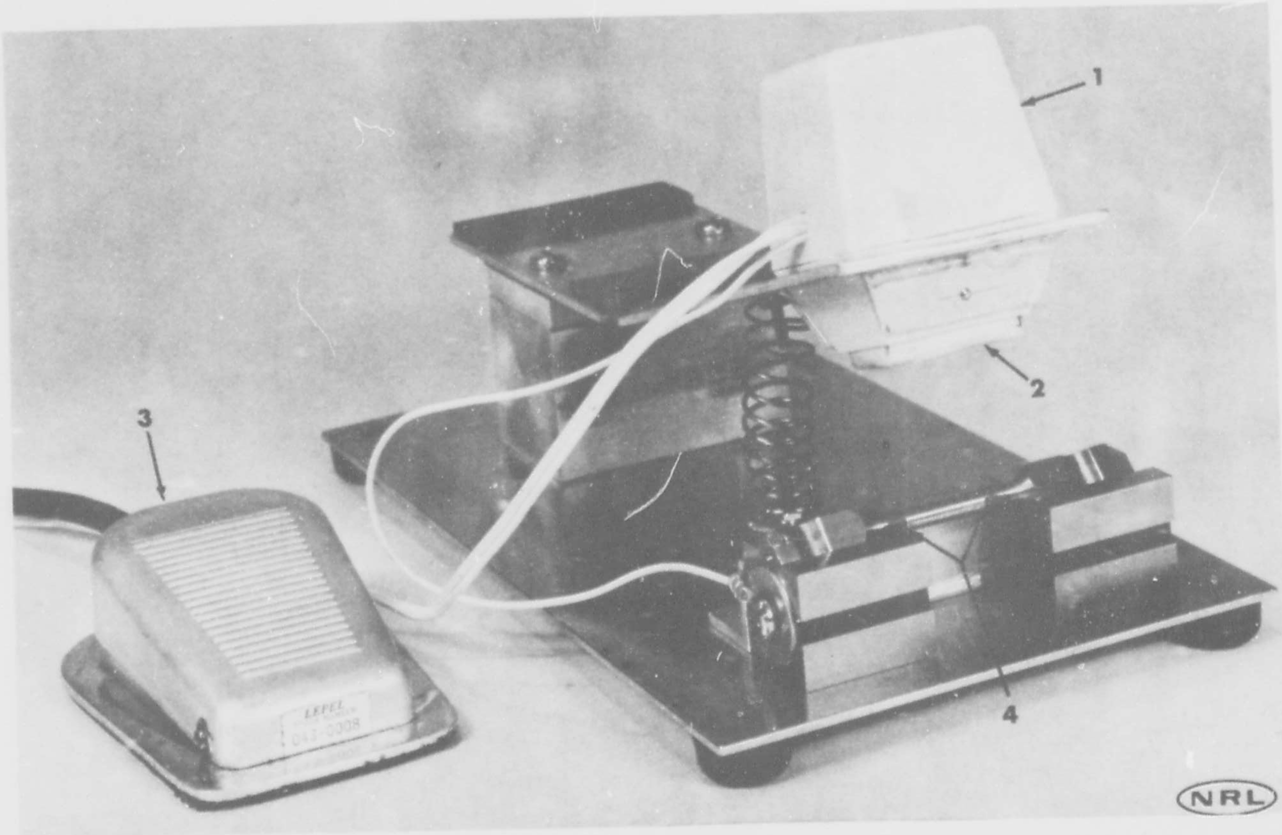

Fig. 8.16 - The electrolytic gage marking device illustrating: (1) etcher, (2) teflon strip over felt pad containing electrolyte to insulate 1-in. section of tensile specimen, (3) footswitch normally positioned outside the cell and interconnected to the etcher via electrical patch panels, and (4) the tensile specimen located in the V-blocks illustrating the marks produced by the etcher

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Office of Naval Research, NAVSHIPS, Atomic Energy Commission Army Nuclear Power Program

13. ABSTRACT

The research program of the NRL Metallurgy Division, Reactor Materials Branch, is devoted to the determination of the effects of nuclear radiation upon the properties of structural materials. The overall program is sponsored by the Office of Naval Research, the Naval Ship Systems Command, the U.S. Atomic Energy Commission, and the Army Nuclear Power Program. Since research findings which apply to the objectives of one sponsoring agency are also of interest to the others, the overall program progress is reported herein. This report, covering research for the period 1 May - 31 July 1967, includes the following: (1) through-thickness radiation resistance of two A533 Grade B, Class I steel plates at 550 F, (2) directional notch ductility performance of irradiated 3-1/2Ni-Cr-Mo and 5Ni-Cr-Mo-V steel plates, (3) radiation sensitivity of A353 (9% nickel) steel as influenced by percent retained austenite, (4) tensile properties behavior versus postirradiation test temperature of selected structural steels, (5) potential for aging embrittlement of pressure vessel steels, (6) postpressurization test operations on PM-2A reactor pressure vessel, and (7) auxiliary equipment developed for elevated temperature remote tension testing of radioactive specimens.

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