NRL Report 6420

# Initial Evaluations of Metallurgical Variables as Possible Factors Controlling the Radiation Sensitivity of Structural Steels

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September 29, 1966

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Washington, D.C.

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# Initial Evaluations of Metallurgical Variables as Possible Factors Controlling the Radiation Sensitivity of Structural Steels

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Abstract: Experimental investigations for the isolation and assessment of metallurgical factors causing variable radiation embrittlement sensitivity of reactor structural steels have been undertaken, using both large-tonnage commercial heats and special laboratory heats of steel. Metallurgical variables being evaluated include the identity and quantity of major alloying elements and of residual elements, steelmaking practice—both melting (refining) and heat treatment practice, microstructure, and gas content.

Experimental results from the initial series of the exploratory screening studies demonstrate that the radiation sensitivity of a steel can be altered appreciably through heat treatment practices and that microstructure plays a dominant, if not the most influential, role in radiation sensitivity development. A tempered martensite structure was noted to be generally less radiation sensitive than tempered upper bainite and ferrite structures. The data also indicate that vacuum melting and the minimization of residual element content yields steels having a superior irradiation performance compared with steels produced by conventional open hearth melting. However, long-term stress relieving heat treatments were not found to alter the irradiation response of A302-B steel.

## INTRODUCTION

The embrittlement of pressure vessel steels by neutron radiation, manifested as an increase in their ductile to brittle transition temperature, is well recognized and accepted as a nuclear service phenomenon (1-5). Further, the establishment of trends in radiation-induced transition temperature increase as a function of neutron exposure (>1 Mev) and irradiation temperature has been possible through the compilation of Charpy-V and drop-weight test data from a large number of materials irradiation experiments (6).

On examining the trend band for <450°F irradiations, shown in Fig. 1, specifically noting the width of the band and the grouping of data points for individual steels within the band, a suggestion of material differences with respect to radiation sensitivity is observed. The existence of significant variability in irradiation response has indeed been confirmed by NRL (7-10) and by the Bettis Atomic Power Division (BAPD) of the Westinghouse Electric Corporation (11) through experiments in which specimens of several steels were irradiated simultaneously to provide matching thermal and nuclear environmental conditions. Using this same technique, heat-to-heat variations in radiation sensitivity as well as dissimilarities in irradiation behavior through the thickness of heavy section plate materials have also been found (12).

Variability in irradiation response, such as that illustrated in Figs. 2 and 3, has greatly stimulated both immediate and long range engineering interest in the isolation of critical metallurgical factors influencing radiation sensitivity. Immediate interest stems from the possibility of controlling metallurgical conditions to achieve consistent sensitivity in a selected steel. Long range interest looks toward establishment of optimum metallurgical conditions which minimize radiation sensitivity and toward the subsequent development of a family of advanced steels having optimum combinations of metallurgical variables for a very low level of embrittlement sensitivity. The realization of this goal may minimize or in a practical sense eliminate embrittlement as a critical factor in reactor design and operation.

In assessing available experimental data at face value, a host of metallurgical factors are found suspect as directly influencing irradiation response, either through singular or combined action. These include the identity and quantity of major alloying constituents and of residual elements, steel making practice—both melting

NRL Problem M01-14; Projects 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 19, 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.

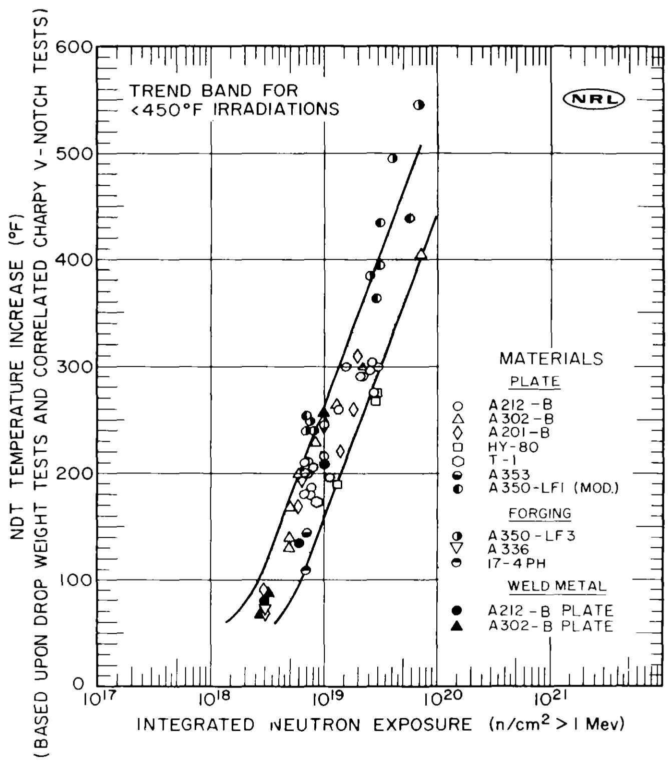



Fig. 1 — Increase in the nil-ductility transition (NDT) temperature of several steels resulting from irradiation at temperatures below 450°F

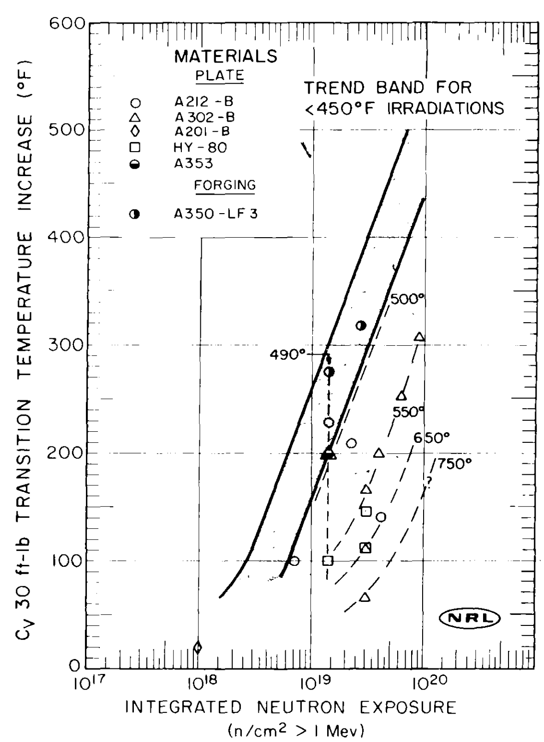



Fig. 2 — Increase in the Charpy-V transition temperatures of several steels resulting from irradiation at temperatures above 450°F. All points on the vertical dashed line reference a single 490°F irradiation experiment in which specimens of five steels were exposed simultaneously.

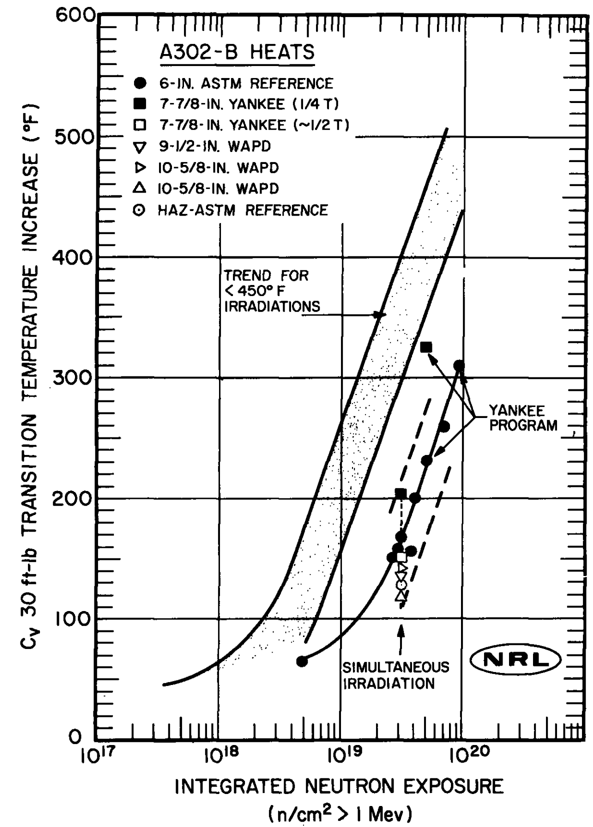



Fig. 3 – Increase in the Charpy-V transition temperatures of several heats of A302-B steel resulting from irradiation at 550°F in the Low Intensity Test Reactor. Six of the eight points on the vertical dashed line reference a single irradiation experiment in which specimens of five steels were exposed simultaneously.

(refining) and heat treatment practice, microstructure, and gas content (O, H, N). To isolate which of these factors are critical, and to assess their influence, experimental programs have been devised and pursued with the close collaboration of the U.S. Steel Corporation, Applied Research Laboratory, using steels from both large-tonnage commercial heats and special laboratory heats. This report describes the several programs undertaken and presents significant research findings to date. Although this is an NRL report, the rate of program progress reported herein was made possible by the direct participation and consultative assistance of the Applied Research Laboratory.

## EXPERIMENTAL APPROACH

Since so many metallurgical variables required assessment, the initial investigations were planned as a primary screening effort to identify the dominant or major factors contributing to radiation sensitivity. With this approach, three general ground rules were established for selecting materials and planning irradiation experiments. First, the singular effects of individual variables would be categorized as dominant, secondary, or noncontributing. Second, specific influences would be ascertained by Charpy-V and tension test methods and evaluations. Third, irradiation exposures would be at low temperatures (<450°F) to eliminate the need for considering complicating thermal "self-annealing" effects which are observed with higher irradiation temperatures and to permit more simple capsule-type irradiation assemblies. Subsequently, the materials were irradiated in the Oak Ridge Low Intensity Test Reactor (LITR) in one of three experimental facilities including the C-18, C-55, and C-43 core lattice positions. A limited number of irradiation experiments were also performed at the Materials Test Reactor (MTR) in the A-4 reflector position. Peak exposure temperatures in all cases were monitored by low melting point alloy detectors, while neutron exposure conditions were established through activation analysis of iron dosimeter wires ($Fe^{54}(n,p)Mn^{54}$ reaction) included in the assemblies.

## IRRADIATION INVESTIGATIONS AND RESULTS

The initial investigations undertaken may be considered as four separate programs, with additional input to the total analysis being gleaned from other concurrent radiation effects studies. The first program was an exploration of the possible alteration of radiation sensitivity through modification of microstructure. The second program involved special heats of A302-B steel and was a qualitative study of the influence of steel melting and heat treatment practices, fabrication (thermal) history, residual elements, and gas content on irradiation response. The third program represented a fully controlled evaluation of nickel content as a sensitivity variable as inferred by earlier experimental results (13,14). The fourth program, a substudy of residual elements in the A302-B steel, was specifically designed to assess the significance of carbon, phosphorus, and sulfur levels to radiation sensitivity but is not discussed in this report, as this investigation is still in progress.

### Program 1: Isolation and Evaluation of the Influence of Microstructure on Radiation Sensitivity

On considering the possible metallurgical factors responsible for differences in radiation sensitivity, previous experimental results strongly suggest that microstructure has a dominant influence. Since other factors may also be contributing to the level of apparent radiation sensitivity, microstructural effects cannot be isolated and verified solely through intercomparisons of response of several different steels. In this study, special precautions were therefore undertaken to insure that all variables except microstructure were maintained constant. The approach involved the reheat treatment of two steels, HY-80 (Ni-Cr-Mo) and A350-LF3, which respectively set the bounds of low and high radiation embrittlement sensitivity depicted by the trend band (Fig. 4). The chemical compositions of these steels are given in Table 1. If the modification

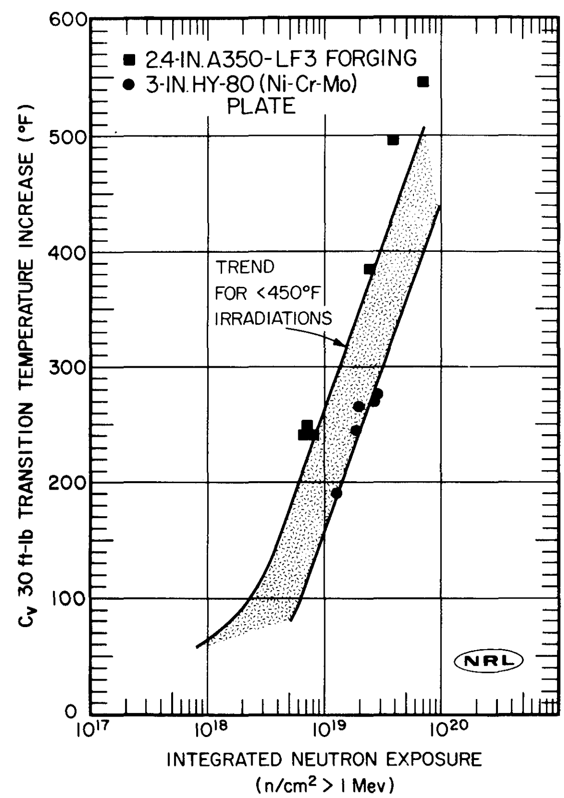


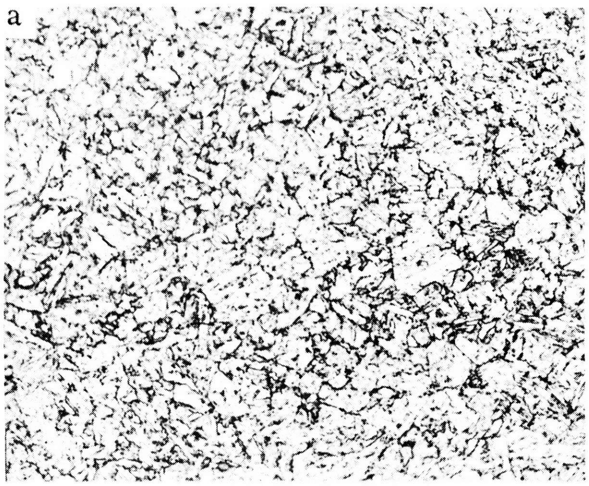

Fig. 4 — Increase in the Charpy-V transition temperatures of HY-80 (Ni-Cr-Mo) and A350-LF3 steels resulting from irradiation at temperatures below 450°F

of the normal microstructures of these steels resulted in a significantly large change or a possible reversal in the order of their relative irradiation response (*i.e.*, the development of high sensitivity in the HY-80 steel and low sensitivity in the A350-LF3 steel), the evidence would prove conclusive as to the contribution of microstructure to radiation sensitivity. Further, the magnitude of differences in irradiation response between the various heat treatment conditions would establish whether or not microstructure has a major or minor influence.

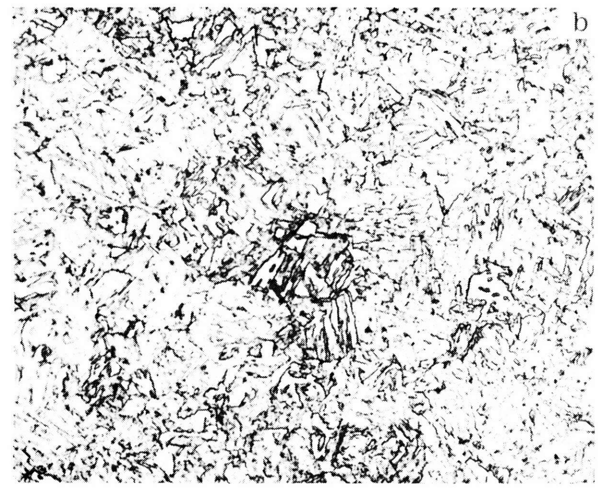
The three reheat treatment schedules employed for each of the two steels, using 1/2-in.-thick plate sections, are described in Table 2. Corresponding microstructures are shown in Figs. 5 and 6. The respective schedules were intended to provide not only a broad range of tensile strengths but also a measure of correspondence in strength level between steels in each reheat-treated condition. From Table 3, it is noted that, in spite of large differences in hardenability and heat treatment response, this objective was largely achieved, with particularly close agreement in tensile strength being obtained in the cases of conditions 1 and 2.

Standard Charpy-V specimens, ASTM Type A (15), from the test sections as well as from the quarter thickness locations of both the 3-in. HY-80 plate and the 2.4-in. A350-LF3 forging, representing the commercially heat treated condition, were irradiated simultaneously at <250°F to a neutron exposure of $2.0 \times 10^{19}$ $n/cm^2$ (>1 Mev) in the LITR. Experimental results for the preirradiated and irradiated conditions are shown in Figs. 7 and 8 and are summarized in Tables 4 and 5.

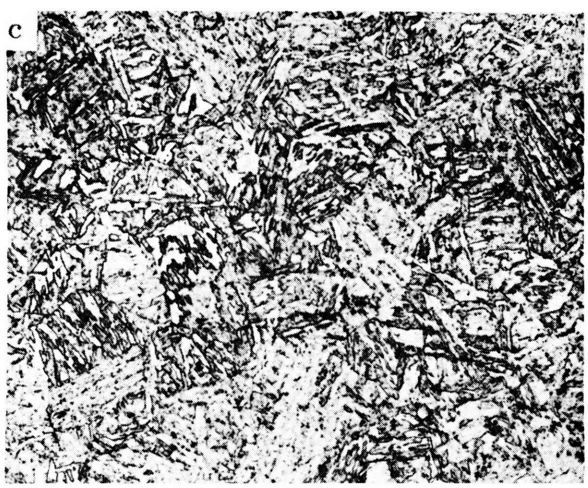
From the data compilations, it is apparent that the radiation sensitivity of both steels can be altered appreciably by heat treatment. In the case of the HY-80 steel (Table 4), the Charpy-V 30-ft-lb transition temperature increases noted for conditions 1, 2, and 3 were all approximately 200°F; condition 4, however, exhibited an increase of 310°F. Comparisons of 50% shear transition temperature increases similarly show a significant difference between condition 4 and conditions 1, 2, and 3. With the A350-LF3 steel, the increases in the Charpy-V 30-ft-lb transition temperature for conditions 2, 3, and 4 were about the same magnitude as were their increase in the



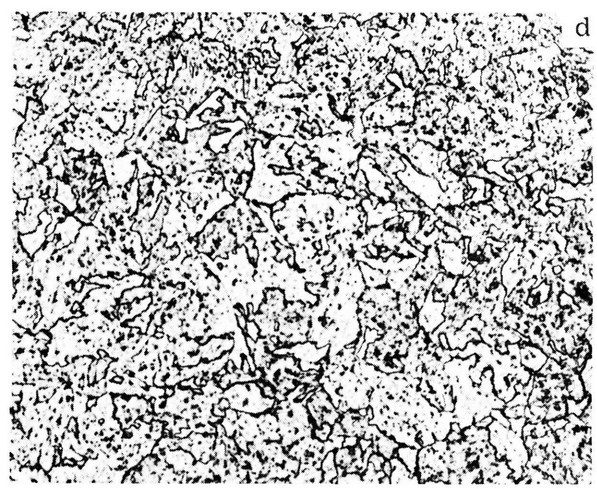
(a) Condition 1: tempered martensite with traces of free ferrite



(b) Condition 2: tempered martensite with traces of free ferrite



(c) Condition 3: (commercial heat treatment) tempered martensite with traces of free ferrite

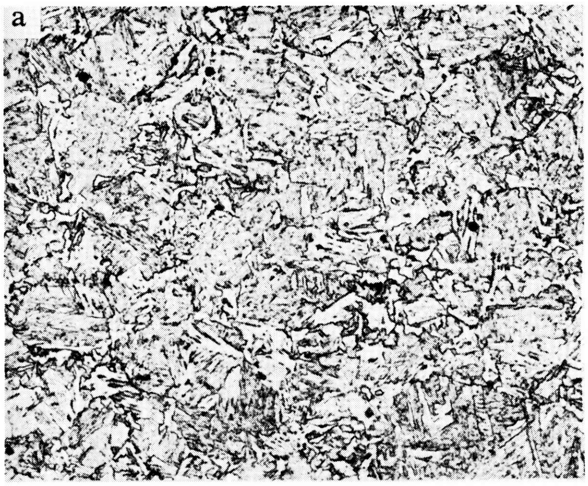


(d) Condition 4: equiaxed ferrite with a uniform distribution of fine carbide particles

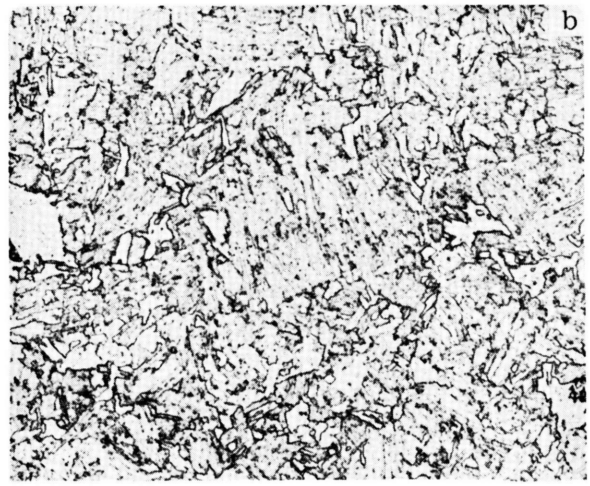
Fig. 5 — Microstructures of reheat treated HY-80 steel (500X)

50% shear transition temperature (~270°F). The 165°F increase in 50% shear transition temperature for condition 1, on the other hand, denotes a much lower radiation sensitivity than that of conditions 2, 3, and 4. The data thus demonstrate that the radiation sensitivity of a normally high response steel (A350-LF3) can be substantially reduced (condition 1) while that of a normally low response steel (HY-80) can be significantly increased (condition 4) through heat treatment practice.

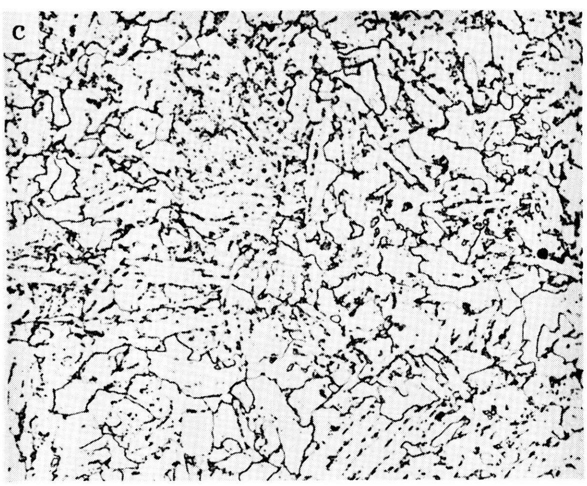
On further evaluating the results of this experiment, it is noted that condition 4 of the HY-80 steel developed a transition temperature increase approximately equal to that for conditions 2, 3, and 4 (high sensitivity) of the A350-LF3 steel. Similarly, equivalent radiation sensitivity, using the 50% shear transition temperature criterion,



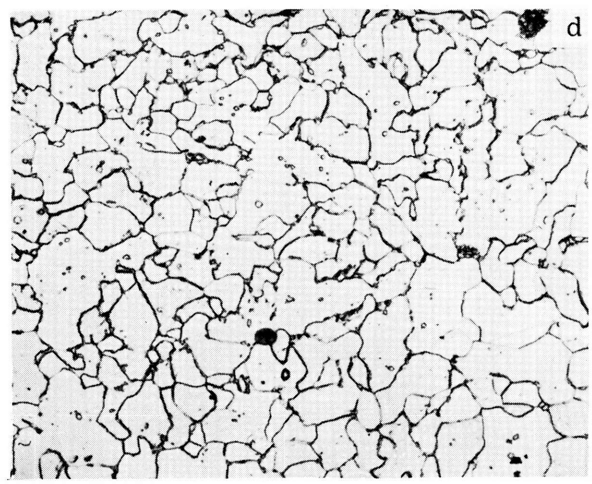
(a) Condition 1: tempered martensite and upper bainite



(b) Condition 2: ferrite containing a uniform distribution of fine carbides and traces of essentially carbon-free ferrite. Vestiges of the original quenched structure are still seen.



(c) Condition 3: (commercial heat treatment) ferrite containing a random distribution of coarsened carbide particles



(d) Condition 4: equiaxed ferrite with an intergranular carbide network

Fig. 6 — Microstructures of reheat treated A350-LF3 steel (500X)

is founa between the response of condition 1 of the A350-LF3 steel and that of conditions 1, 2, and 3 (low sensitivity) of the HY-80 steel. Since the limits of the NRL trend band for <450°F irradiations were for the most part established by data for these two steels in the commercially heat treated condition, it becomes apparent that microstructure has a highly dominant influence on radiation sensitivity.

On relating the experimental results to the microstructure of the respective material heat treatment conditions, a pattern of microstructure *versus* radiation behavior emerges. As shown in Fig. 5, the structures of the HY-80 steel in conditions 1, 2, and 3 are all typical of tempered martensite with traces of free ferrite. This similarity of structures is consistent with the equivalence of irradiation response noted. The

TABLE 1
Chemical Composition and Commercial Heat Treatment of HY-80 and A350-LF3 Steels

| Steel | Form | Thick-ness (in.) | Chemical Analysis (wt-%) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | C | Mn | P | S | Si | Ni | Cr | Mo | Al | V |
| HY-80 (Ni-Cr-Mo) | Plate* | 3 | 0.14 | 0.21 | 0.011 | 0.014 | 0.19 | 2.91 | 1.55 | 0.54 | 0.06 | 0.04 |
| A350-LF3 | Forging† | 2.4 | 0.14 | 0.52 | 0.031 | 0.032 | 0.25 | 3.28 | 0.04 | 0.05 | – | 0.04 |

*Heat treatment: austenitized at 1650°F for 3 hours; water quenched; tempered at 1175°F for 3 hours; air cooled.
†Heat treatment: forged in the temperature range 1700°F minimum to 2250°F maximum.
Post-forging treatment: normalized at 1550°F; water quenched; tempered at 1200°F; stress relieved at 1150°F.

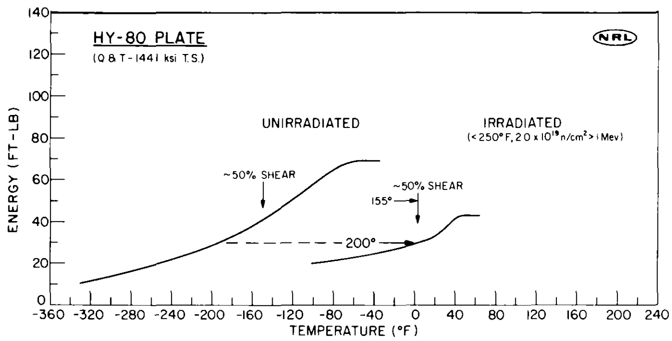
TABLE 2
Reheat-Treatment Schedules for HY-80 and A350-LF3 Steels

| Condition | Desired Tensile Strength (ksi) | Heat Treatment |
|---|---|---|
| | | HY-80 Steel |
| 1 | 135/145 | Austenitized at 1550°F for 1 hour; water quenched; tempered at 1050°F for 1 hour; water quenched (this heat treatment designated Q&T-1 in subsequent tables). |
| 2 | 100/105 | Austenitized at 1550°F for 1 hour; water quenched; tempered at 1225°F for 1 hour; water quenched (Q&T-2). |
| 3 | – | As commercially heat treated. |
| 4 | Less than commercial | Normalized at 1650°F for 1 hour; air cooled; recharged into 1550°F furnace; held 1 hour; air cooled to 1050°F; recharged into 1050°F furnace; held at 1050°F for 48 hours; heated to 1200°F; held at 1200°F for 24 hours; furnace cooled to room temperature. |
| | | A350-LF3 Steel |
| 1 | 135/145 | Austenitized at 1550°F for 1 hour; water quenched; tempered at 650°F for 1/2 hour; water quenched (Q&T-1). |
| 2 | 100/105 | Austenitized at 1550°F for 1 hour; water quenched; tempered at 1125°F for 1 hour; water quenched (Q&T-2). |
| 3 | – | As commercially heat treated. |
| 4 | Less than commercial | Austenitized at 1550°F for 1 hour; furnace cooled to 1200°F in 24 hours (15°F/hr); furnace cooled to 600°F in 12 hours (50°F/hr); air cooled. |

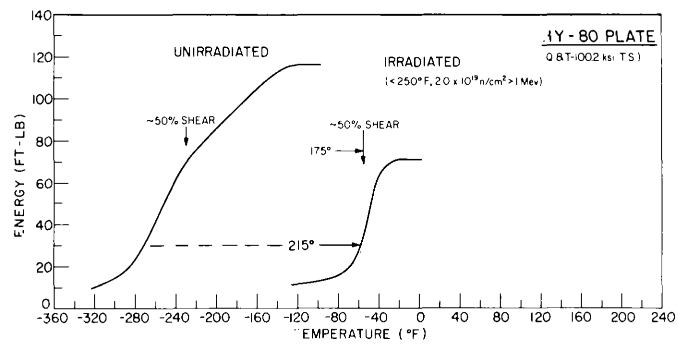
TABLE 3
Yield and Tensile Strengths of Reheat-Treated HY-80 and A350-LF3 Steels

| Condition | Heat Treatment | HY-80 | | A350-LF3 | |
|---|---|---|---|---|---|
| | | Yield* Strength (psi) | Tensile Strength (psi) | Yield* Strength (psi) | Tensile Strength (psi) |
| 1 | Q&T-1 | 134,750 | 144,150 | 115,100 | 137,550 |
| 2 | Q&T-2 | 85,800 | 100,250 | 94,650 | 103,450 |
| 3 | Commercial | 84,800 | 100,050 | 58,575 | 82,150 |
| 4 | Austenitized; furnace cooled | 66,550 | 84,600 | 42,300 | 71,500 |

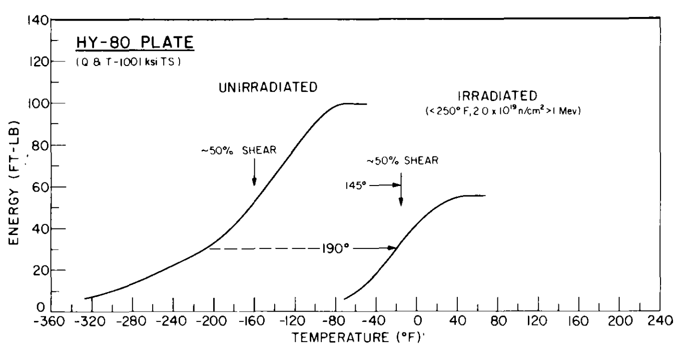
*0.2% offset yield strength, 0.252-in.-gage-diameter specimens.



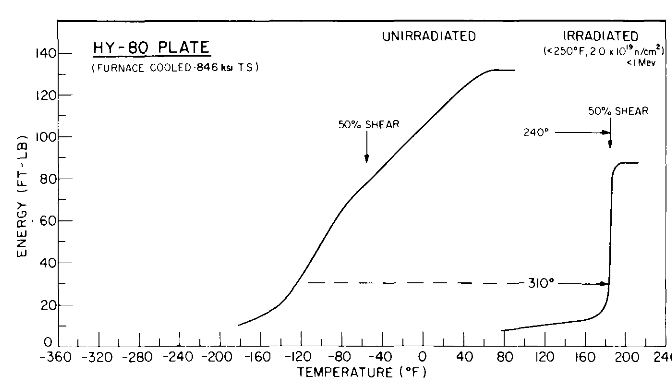

(a) Quenched and tempered, condition 1 (see Table 2)




(b) Quenched and tempered, condition 2

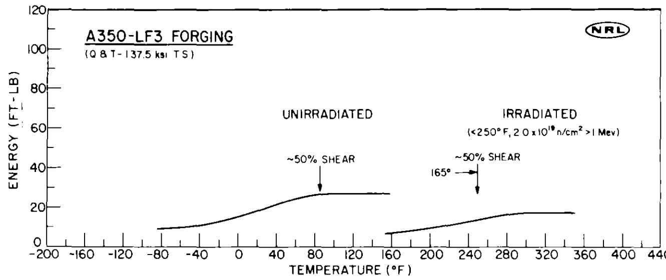



(c) Commercially heat treated, condition 3

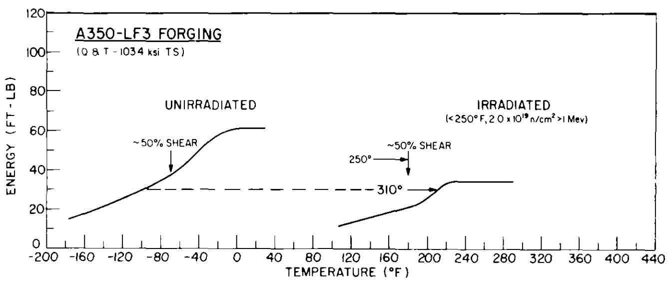



(d) Austenitized and furnace cooled, condition 4

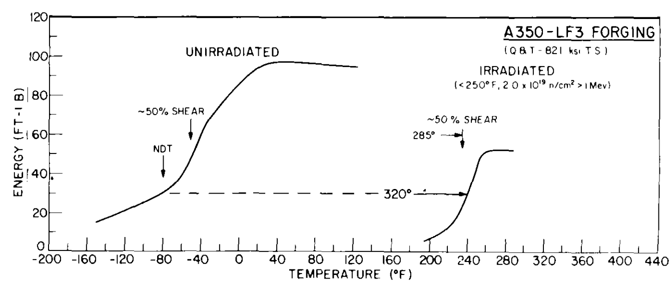
Fig. 7 – Notch ductility behavior of HY-80 (Ni-Cr-Mo) plate before and after irradiation at <250°F to 2.0 × $10^{19}$ n/cm² (>1 Mev)



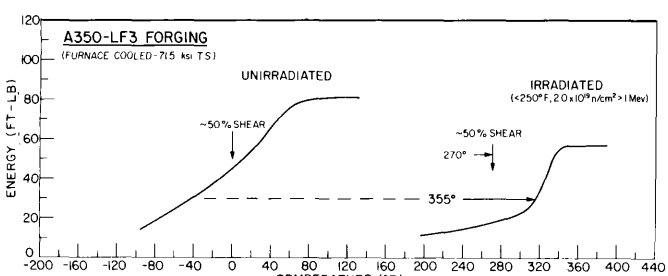

(a) Quenched and tempered, condition 1




(b) Quenched and tempered, condition 2




(c) Commercially heat treated, condition 3




(d) Austenitized and furnace cooled, condition 4

Fig. 8 — Notch ductility behavior of A350-LF3 forging before and after irradiation at <250°F to $2.0 \times 10^{19}$ n/cm$^2$ (>1 Mev)

TABLE 4
Charpy-V Transition Temperature Behavior of HY-80 Steel in Various Heat Treatment Conditions Before and After Irradiation at <250°F to 2.0 × $10^{19}$ n/$cm^2$ (>1 Mev)

| Condition | Heat Treatment | Charpy-V 30-ft-lb Transition Temperature (°F) | | | Charpy-V 50% Shear Transition Temperature (°F) $\Delta T$ |
|---|---|---|---|---|---|
| | | Initial | Final | $\Delta T$ | |
| 1 | Q&T-1 | −195 | 5 | 200 | 155 |
| 2 | Q&T-2 | −270 | −55 | 215 | 175 |
| 3 | Commercial | −210 | −20 | 190 | 145 |
| 4 | Austenitized; furnace cooled | −125 | 185 | 310 | 240 |

TABLE 5
Charpy-V Transition Temperature Behavior of A350-LF3 Steel in Various Heat Treatment Conditions Before and After Irradiation at <250°F to 2.0 × $10^{19}$ n/$cm^2$ (>1 Mev)

| Condition | Heat Treatment | Charpy-V 30-ft-lb Transition Temperature (°F) | | | Charpy-V 50% Shear Transition Temperature (°F) $\Delta T$ |
|---|---|---|---|---|---|
| | | Initial | Final | $\Delta T$ | |
| 1 | Q&T-1 | —* | —* | — | 165 |
| 2 | Q&T-2 | −100 | 210 | 310 | 250 |
| 3 | Commercial | − 80 | 240 | 320 | 285 |
| 4 | Austenitized; furnace cooled | − 40 | 315 | 355 | 270 |

*Full shear energy absorption less than 30 ft-lb.

microstructure of condition 4, however, is quite different, consisting of equiaxed ferrite with a uniform distribution of fine carbides, which accounts for its departure from the general level of sensitivity shown by conditions 1, 2, and 3. Similarly, the major difference in microstructural appearance between the four heat treatment conditions of the A350-LF3 steel (Fig. 6) is found between condition 1 and conditions 2, 3, and 4. Again, this difference is consistent with the pattern of the experimental test data.

Three general observations may thus be listed. First, the radiation sensitivity of steels can be altered appreciably by heat treatment procedures. Second, microstructure is a dominant factor in the radiation sensitivity exhibited by a steel. Third, it would appear that a quenched and tempered structure such as tempered martensite has a lower radiation sensitivity than that of higher temperature transformation products such as ferrite.

### Program 2: Contribution of Steel Melting (Refining) Practice, Heat Treatment History, and Residual Element Level to Radiation Sensitivity

The program for the assessment of steel melting (refining) practice, heat treatment history, and residual element level as radiation sensitivity variables required the casting of special laboratory heats in order to achieve the desired range of material conditions. The base composition selected for study was that of ASTM A302-B steel, as a

Table 6
Chemical Composition Specifications of A302-B Steels for Determining the Effect of Impurities and Residuals on Radiation-Induced Embrittlement

| Heat | Chemical Analysis (wt-%) | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | Mn | P | S | Si | Ni | Cr | Mo | Cu | Ti | Sn | Ta | N | Al |
| Commercial ASTM A302B reference heat (No. A0421) | 0.24 | 1.34 | 0.011 | 0.023 | 0.23 | 0.18 | 0.11 | 0.51 | 0.20 | 0.015 | 0.037 | 0.02 | 0.008 | 0.04 |
| 300-lb air induction heat with residuals | 0.22/0.26 | 1.30/1.40 | 0.010/0.015 | 0.020/0.025 | 0.20/0.25 | 0.15/0.20 | 0.10/0.15 | 0.50/0.55 | 0.18/0.22 | 0.01/0.03 | 0.02/0.04 | 0.02/0.04 | 0.006/0.010 | 0.03/0.05 |
| 300-lb air induction heat without residuals | 0.22/0.26 | 1.30/1.40 | 0.005 max | 0.005 max | 0.20/0.25 | 0.05 max | 0.05 max | 0.50/0.55 | 0.01 max | 0.005 max | 0.005 max | 0.005 max | 0.004 max | 0.03/0.05 |
| 300-lb vacuum induction heat without residuals | 0.22/0.26 | 1.30/1.40 | 0.005 max | 0.005 max | 0.20/0.25 | 0.05 max | 0.05 max | 0.50/0.55 | 0.01 max | 0.005 max | 0.005 max | 0.005 max | 0.004 max | 0.03/0.05 |

large variation in irradiation response was noted previously with heavy section plates from five different commercial heats of this steel (Fig. 3). Following the specifications listed in Table 6, three 300-lb laboratory heats were melted. The ingots obtained were broken down into 1/2-in.-thick plates using a nominal 1:1 cross rolling ratio. The required materials inventory for the program was completed with the addition of 5/8-in. plate sections from the 6-in. ASTM A302-B steel plate selected as a control or reference standard. A 1/2-in. plate from one additional 300-lb vacuum induction heat (with no residuals) having 1.35 wt-% nickel and <0.01 percent manganese was also included in one phase of the program to study the effects of a nickel substitution for manganese. The chemistry of each steel is given in Table 7.

Table 8 illustrates the scope of the program and the extent of possible crosscorrelations and evaluations regarding the variables investigated. Through the intercomparison of experimental results for the commercial heat and the three laboratory heats, the effects of residual element (P, S, Ni, Cr, Cu, Sn, *etc.*) levels and melting procedures (open hearth, air induction, vacuum induction) could be evaluated. For example, the residual element content of heat 2 was matched to that of heat 1 to permit the comparison of air induction *versus* open hearth melting practices. With the placement of each of the four heats in seven heat treatment conditions depicting four general strength categories, the effects of heat treatment procedures, *i.e.,* thermal history, and associated microstructure could be ascertained.

As 28 combinations of materials and heat treatment conditions (excluding the nickel-bearing heat) were involved, a division of materials into several irradiation experiments was required. The natural grouping of materials into four strength conditions listed in Table 8 formed the basis for this division. In addition, experimental assembly space limitations necessitated separate irradiations of Charpy-V and tension test specimens. To simplify the analysis of the experimental results, data pertaining to each strength condition are presented individually.

*Condition 1: Heat Treatment Conditions Representative of Commercial Practices*—Initial Charpy-V 30-ft-lb transition temperatures of the laboratory

TABLE 7
Chemical Composition of A302-B Commercial Reference Heat and Special 300-lb Laboratory Heats

| Heat | Analysis Source | Chemical Analysis (wt-%) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | C | Mn | P | S | Si | Ni | Cr | Mo | Cu | Ti | Sn | Ta | Al | V | N |
| Commercial heat | NRL | 0.23 | 1.35 | 0.015 | 0.021 | 0.22 | 0.22 | 0.12 | 0.52 | 0.12 | 0.02 | ≤0.01 | 0.02 | 0.02 | ≤0.01 | — |
| | U.S. Steel | 0.24 | 1.34 | 0.011 | 0.023 | 0.23 | 0.18 | 0.11 | 0.51 | 0.20 | — | 0.037 | — | 0.038 | — | 0.008 |
| Air induction heat with residuals | NRL | 0.24 | 1.30 | 0.006 | 0.018 | 0.28 | 0.24 | 0.18 | 0.55 | 0.14 | 0.02 | 0.03 | 0.02 | 0.02 | 0.02 | — |
| | U.S. Steel | 0.24 | 1.37 | 0.014 | 0.019 | 0.29 | 0.18 | 0.18 | 0.53 | 0.19 | 0.017 | 0.028 | 0.015 | 0.045 | 0.007 | 0.007 |
| Air induction heat without residuals | NRL | 0.25 | 1.29 | 0.001 | 0.004 | 0.30 | 0.06 | 0.08 | 0.55 | 0.03 | ≤0.01 | ≤0.01 | <0.01 | 0.02 | ≤0.01 | — |
| | U.S. Steel | 0.28 | 1.32 | 0.004 | 0.004 | 0.33 | 0.036 | 0.053 | 0.52 | <0.005 | <0.005 | 0.005 | 0.002 | 0.041 | <0.005 | 0.004 |
| Vacuum melted heat without residuals | NRL | 0.24 | 1.35 | 0.001 | 0.008 | 0.22 | 0.05 | 0.08 | 0.55 | — | — | — | — | 0.02 | — | — |
| | U.S. Steel | 0.24 | 1.31 | 0.005 | 0.005 | 0.25 | 0.021 | 0.003 | 0.52 | <0.005 | <0.005 | — | — | 0.043 | 0.005 | 0.001 |
| Vacuum melted, nickel-bearing heat | NRL | 0.24 | 0.005 | 0.001 | 0.005 | 0.22 | 1.35 | 0.03 | 0.55 | — | — | — | — | 0.02 | — | — |
| | U.S. Steel | 0.24 | 0.01 | 0.004 | 0.004 | 0.24 | 1.39 | 0.005 | 0.50 | <0.005 | <0.005 | — | — | 0.044 | <0.005 | 0.001 |

TABLE 8
Scope of the Experimental Program (Program 2)
Involving Special A302-B Laboratory Heats

| Variables Investigated | Steel Heats and Heat Treatment Conditions |
|---|---|
| Residual elements and melting practice | Heat 1: commercial (reference) heat<br>Heat 2: air induction heat *with* residuals<br>Heat 3: air induction heat *without* residuals<br>Heat 4: vacuum induction heat *without* residuals |
| Heat treatment and microstructure | Condition 1: commercial practice<br>As fabricated<br>As fabricated plus stress relieved<br>Condition 2: high strength condition<br>Quenched and tempered<br>Quenched, tempered, and stress relieved<br>Condition 3: low strength condition<br>Slow cooled after austenitizing<br>Condition 4: intermediate strength condition<br>Quenched and tempered<br>Quenched, tempered, and stress relieved |

heats and of the reference heat in the "commercially heat treated" condition are presented in Table 9. As noted in Table 10, special heat treatment procedures were employed for the 1/2-in. plates of the laboratory heats to develop properties and microstructures "matching" the quarter thickness characteristics of the 6-in. commercially heat treated reference steel. Thus, the differences noted in Charpy-V 30-ft-lb transition temperature behavior in the "austenitized and tempered" condition are not unaccounted for and, further, are not unrealistic considering the differences in section sizes involved. With reference to the stress relieved condition (six stress relief cycles at 1125°F for 30 hours total time at temperature) and initial transition temperatures listed in Table 9, no appreciable change in notch ductility characteristics of the commercial reference heat or the air induction heat with matching residuals was observed with stress relieving. However, this stress relief did increase the transition temperatures of the remaining heats. As noted in Table 11, some loss

TABLE 9
Charpy-V 30-ft-lb Transition Temperature Behavior of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats After Irradiation at <250°F to 3.1 × $10^{19}$ n/cm² (>1 Mev)

| Heat | Heat Treatment Condition | Charpy-V 30-ft-lb Transition Temperature (°F) | | |
|---|---|---|---|---|
| | | Initial | Irradiated | $\Delta T$ |
| Commercial heat | Austenitized and tempered | 25 | 335 | 310 |
| | Stress relieved* | 30 | 345 | 315 |
| Air induction heat with residuals | Austenitized and tempered | 65 | 350 | 285 |
| | Stress relieved* | 60 | 340 | 280 |
| Air induction heat without residuals | Austenitized and tempered | −35 | 235 | 270 |
| | Stress relieved* | 5 | 245 | 240 |
| Vacuum melted heat without residuals | Austenitized and tempered | 55 | 240 | 185 |
| | Stress relieved* | 145 | 340 | 195 |
| Vacuum melted, nickel-bearing heat† | Austenitized and tempered | 70 | 340 | 270 |
| | Stress relieved* | 145 | 395 | 250 |

*Six stress relief cycles at 1125°F for 30 hours total.
†Special heat with 1.35 wt-% nickel and 0.01 wt-% manganese.

TABLE 10
Heat Treatment of the Special 300-lb Laboratory Heats for Simulation of the Standard Heat Treatment Condition

| Heat | Plate Thickness (in.) | Heat Treatment |
|---|---|---|
| Commercial heat | 6 | Austenitized at 1650°F for 2 hours; water quenched; tempered at 1200°F for 6 hours; furnace cooled to below 600°F |
| Laboratory heats | 1/2 | Austenitized at 1650°F for 1 hour; air cooled; tempered at 1200°F for 1 hour; water quenched |

TABLE 11
Tensile Properties of the A302-B Reference Heat and of the 300-lb Laboratory Heats Representing Commercial Practice

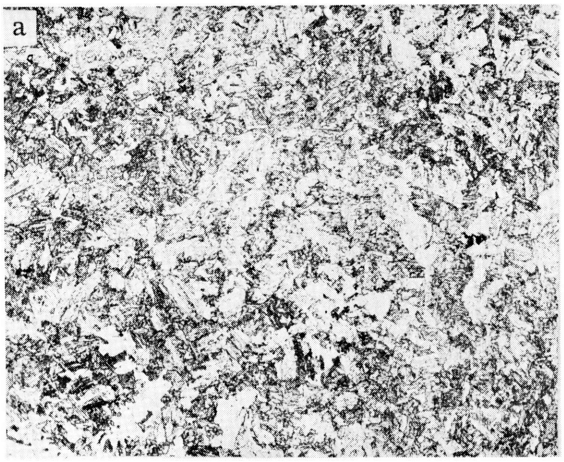
| Heat | Yield Strength (0.2% Offset)* (ksi) | | Tensile Strength† (ksi) | |
|---|---|---|---|---|
| | As Tempered | Stress Relieved | As Tempered | Stress Relieved |
| Commercial heat | 65.5 | 62.4 | 87.9 | 83.9 |
| Air induction heat with residuals | 80.8 | 73.4 | 98.6 | 92.2 |
| Air induction heat without residuals | 70.1 | 64.4 | 90.4 | 86.2 |
| Vacuum melted heat without residuals | 73.7 | 67.7 | 93.3 | 87.1 |
| Vacuum melted, nickel-bearing heat | 68.7 | 64.5 | 86.1 | 82.4 |

*0.252-in.-gage-diameter specimens.
†Average values from three specimen tests.

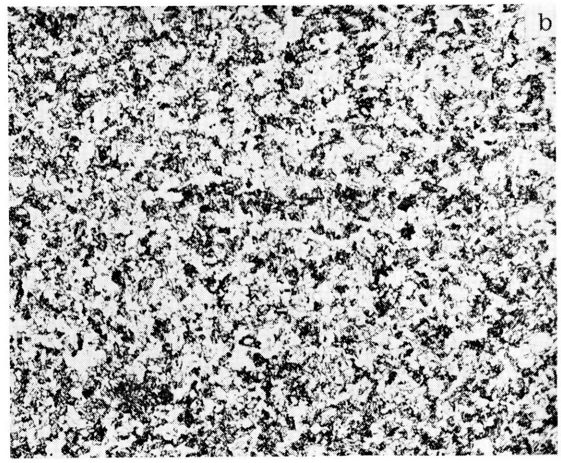
in yield (0.2 percent offset) and tensile strength was observed for all heats. Microstructural observations related to the development of these increases are discussed in a later section.

For examinations of irradiation response, specimens of the laboratory heats, including the nickel-bearing heat, and of the commercial reference heat, were irradiated simultaneously at <250°F to $3.1 \times 10^{19}$ n/cm² (>1 Mev). From the experimental results summarized in Table 9, four general observations were made. First, stress relieving was not found to alter irradiation response ($\Delta T$) even though initially producing an increase in the preirradiation transition temperature in some cases. Second, the irradiation embrittlement sensitivity of the air induction heat *with* residuals was about equivalent to that of the air induction heat *without* residuals, suggesting that residual element level is a noninfluential factor. Third, the vacuum melted heat exhibited a lower transition temperature increase than either of the air induction heats or the commercial heat, indicating an appreciable contribution of gas content to radiation sensitivity. Finally, the transition temperature increase of the vacuum melted nickel-containing heat was much greater than that of the vacuum melted A302-B heat, being essentially equivalent to that of both air induction heats. As will be demonstrated in a later section, however, the higher irradiation response of the nickel-bearing heat *versus* the vacuum melted A302-B heat most probably is not a *direct* influence of nickel content on embrittlement sensitivity.

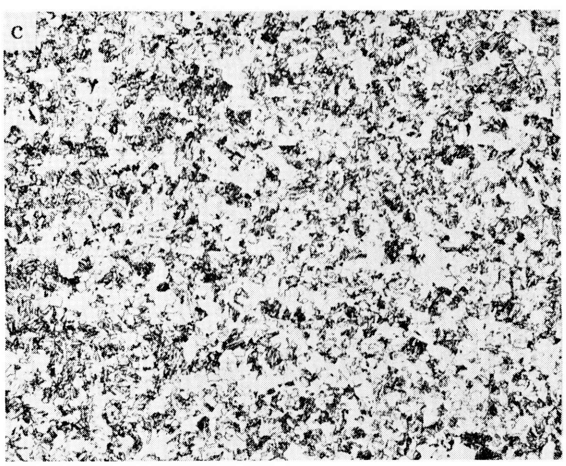
Microstructures for both the austenitized and tempered and the austenitized, tempered, and stress relieved conditions of the individual steels in this series are presented in Figs. 9, 10, and 11. As noted from Figs. 9 and 10, each of the matrices can be generally classified as tempered upper bainite. However, the structures are not fully comparable in each case from the standpoint of the size as well as appearance of the formations. Rather, they fall into two groupings with one group consisting of both air induction heats and the vacuum melted nickel bearing heat, and the other group consisting of the commercial reference heat and the vacuum melted A302-B heat. Within the second group, a further distinction between the reference and vacuum melted heats can be made in that the structure of the vacuum melted heat appears more equiaxed with less overall uniformity in the carbide distribution. Before comparing the microstructures for the as tempered and stress relieved conditions on an individual basis, small microstructural differences would be expected in view of the data presented in Table 9 for the unirradiated condition.



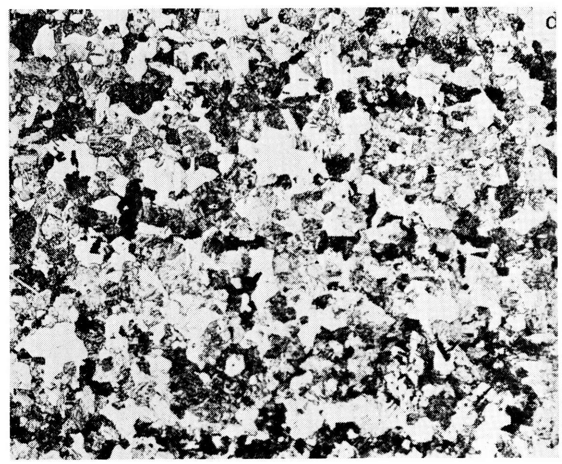

(a) Commercial heat



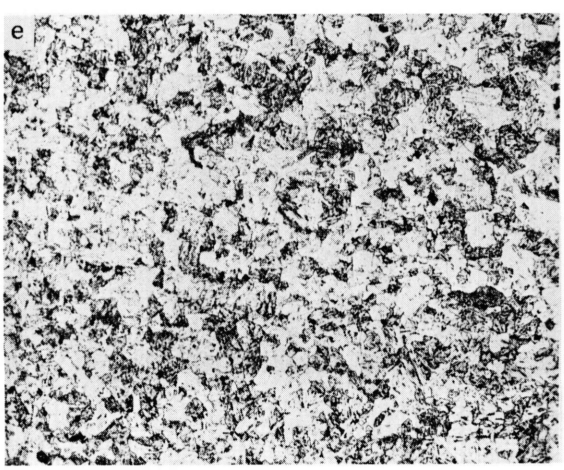

(b) Air induction heat with residuals




(c) Air induction heat without residuals

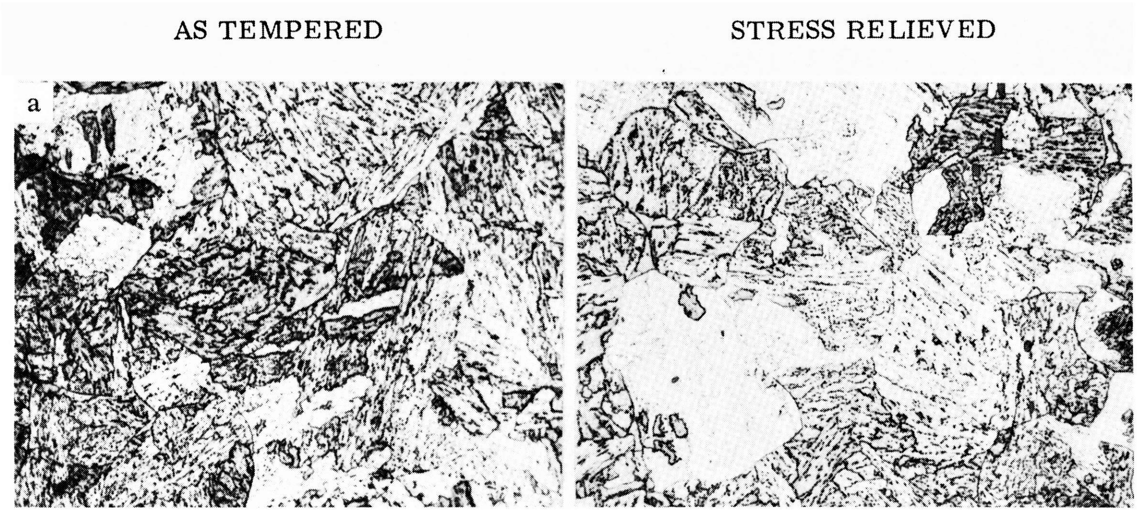



(d) Vacuum melted heat without residuals

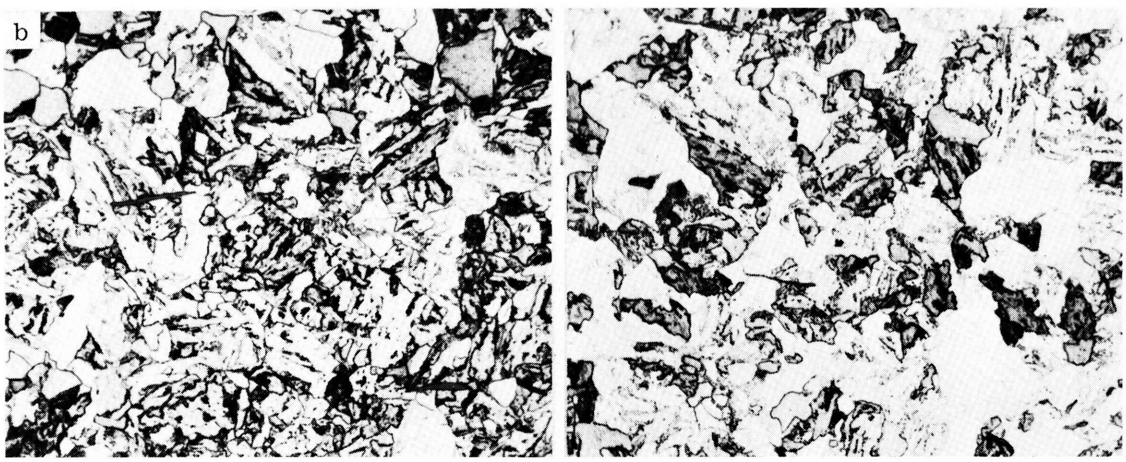



(e) Vacuum melted, nickel bearing heat

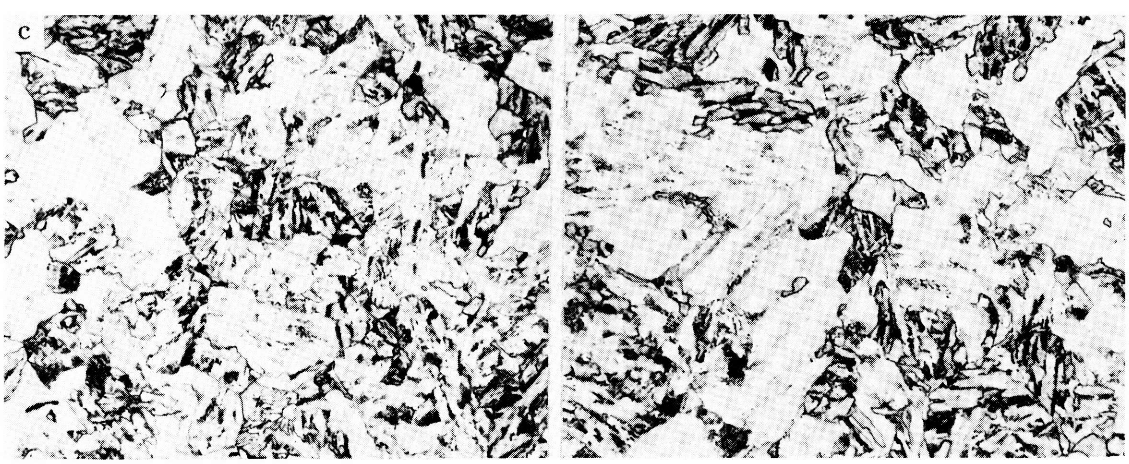
Fig. 9 — Microstructures of the A302-B commercial reference heat and of the laboratory heats in the "commercially tempered" condition. Tempered upper bainite (100X).



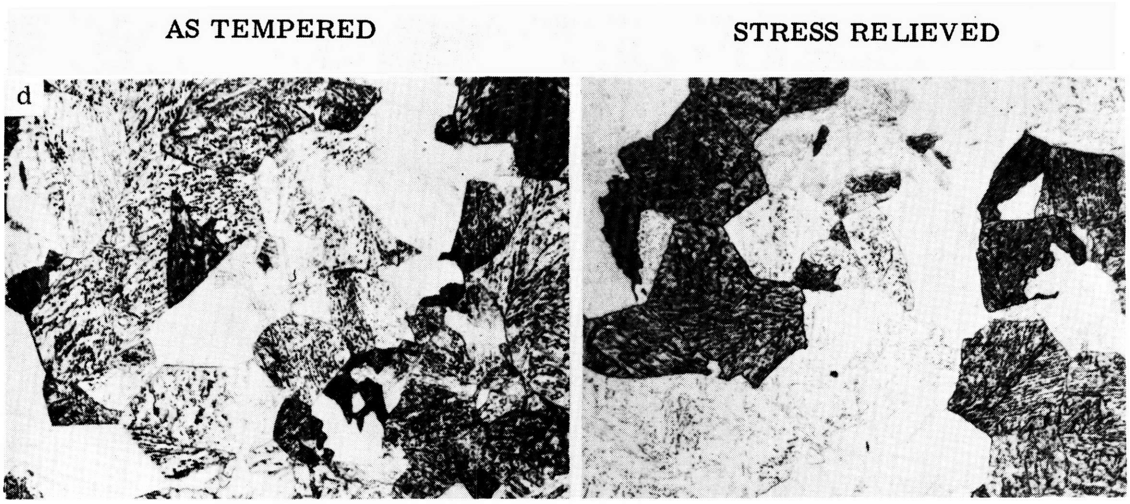

(a) Commercial heat



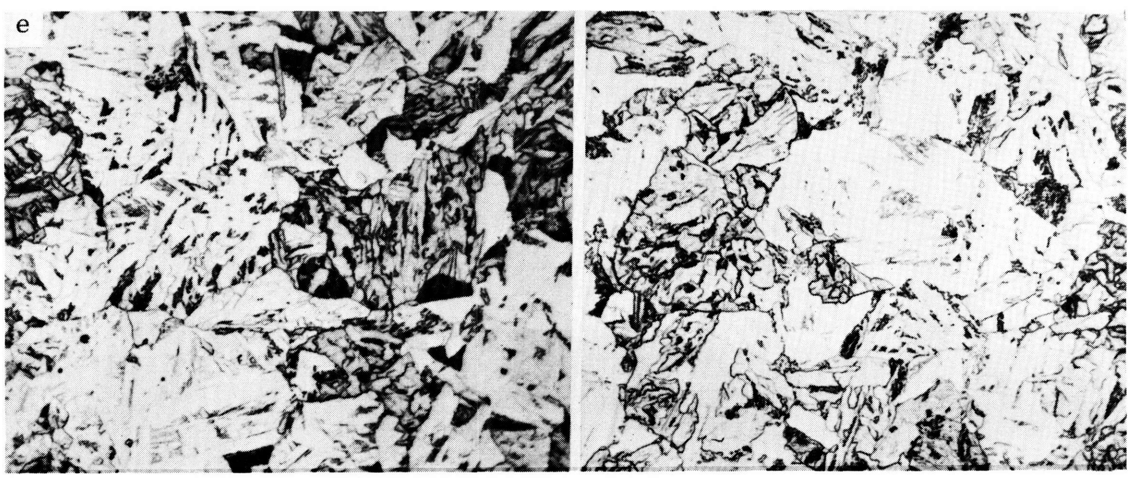

(b) Air induction heat with residuals




(c) Air induction heat without residuals

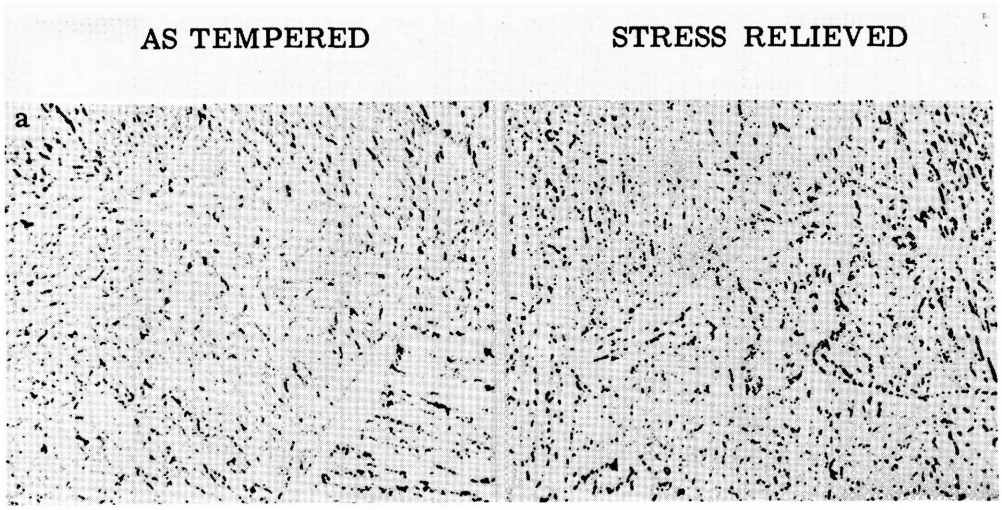



(d) Vacuum melted heat without residuals

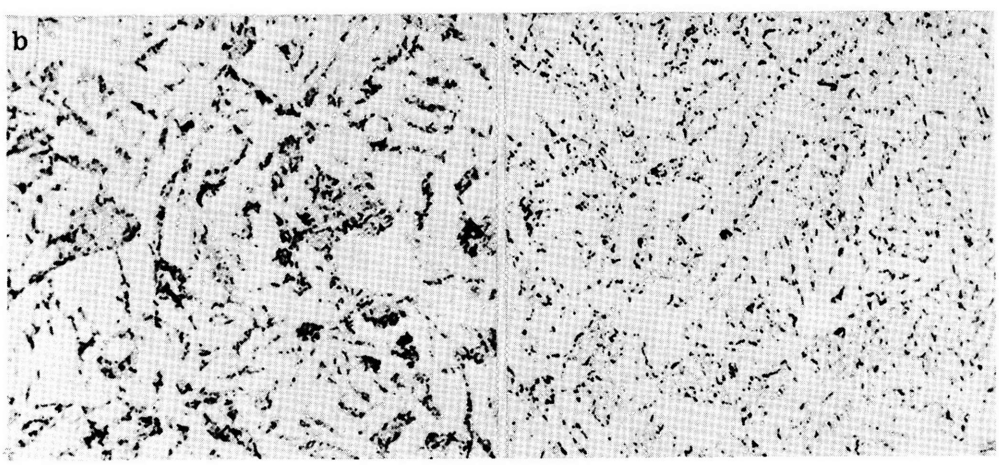



(e) Vacuum melted, nickel bearing heat

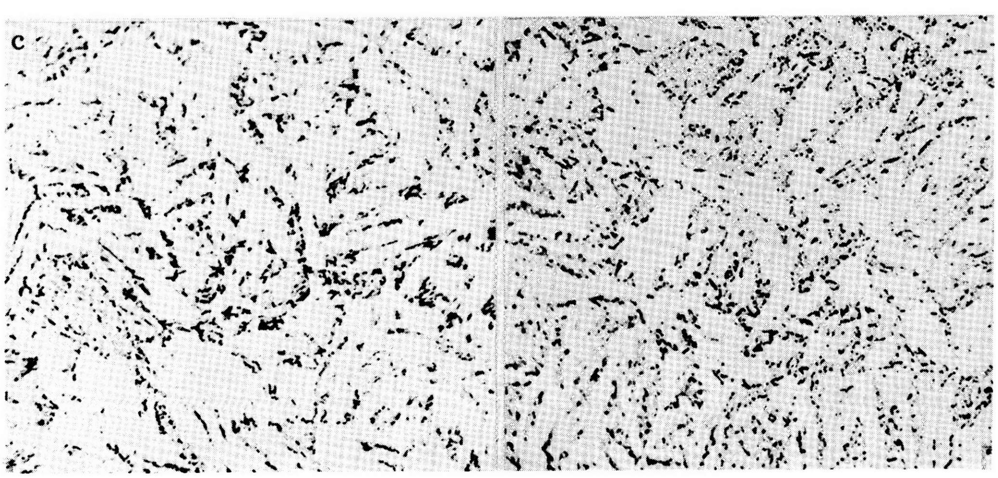
Fig. 10 – Microstructures of the A302-B commercial reference heat and of the laboratory heats in the "commercially heat treated" condition. Tempered upper bainite (500X).



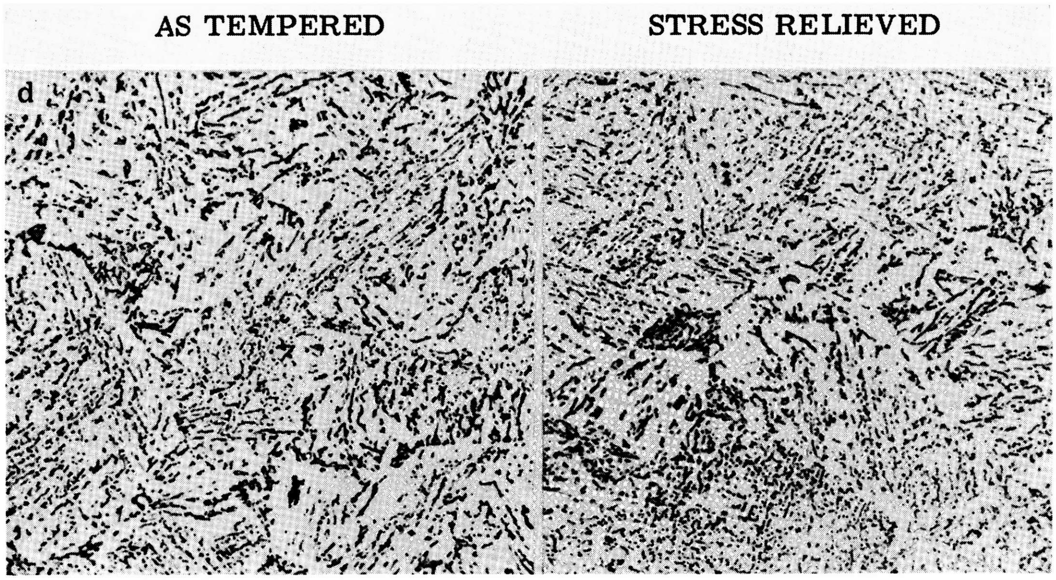

(a) Commercial heat



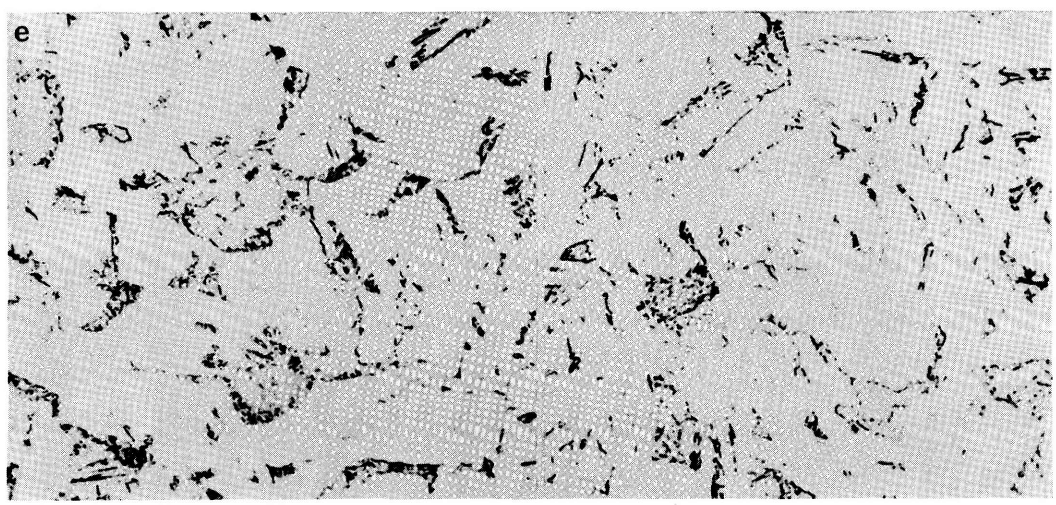

(b) Air induction heat with residuals




(c) Air induction heat without residuals




(d) Vacuum melted heat without residuals




(e) Vacuum melted, nickel bearing heat

Fig. 11 — Microstructures of the A302-B commercial reference heat and of the laboratory heats in the "commercially heat treated" condition showing carbide size and distribution (Super Picral Etchant; 1000X). (Courtesy of U.S. Steel Applied Research Laboratory.)

These differences are not readily seen from Figs. 9 and 10 but are found on examining the nature and distribution of the carbides at higher magnification (Fig. 11) and are consistent with transition temperature increases noted with the long-term (30-hour) stress relieving treatment.

The general observations on irradiation response, listed earlier, did not consider possible microstructural effects in the demonstrated behavior patterns. However, the inclusion of microstructural evidence in the analyses serves to reinforce the validity of the observations made. Although some differences in matrix appearance were found among the different heats, the grouping of the structures noted proved conducive to the analysis. The microstructural differences observed may indeed be considered fortuitous as permitting the coarse evaluation of two other possible sensitivity factors: prior austenite grain size and carbide distribution. The structures shown in Fig. 9 coupled with the experimental data, for example, are not suggestive of an influence of prior austenite grain size. On the other hand, the increase in "carbide free" areas noted on comparing the matrices of the reference heat *versus* the air induction heats *versus* the vacuum melted heat, paralleling the decrease in radiation sensitivity, may be indicative of a subtle role of carbide distribution in the level of irradiation response.

*Condition 2: High Strength Condition*—The two heat treatment conditions employed for comparisons of the relative irradiation responses of the laboratory heats and the commercial reference heat at strength levels considerably above that normally specified for A302-B steel are given in Table 12. Tensile properties developed by the quench and tempering treatments are listed in Table 13. Microstructures of the steels are compared in Fig. 12. As with the "commercially heat treated" steel series above, a grouping of these steels with relation to structural appearances was noted. However, the grouping was not the same; in this case the structures of the air induction heat *with* residuals matched that of the reference heat, while those of the air induction heat *without* residuals more closely resembled the structures of the vacuum melted heat. The nickel-bearing vacuum melted heat was not included in this investigation or in the studies of the low strength condition to follow because of irradiation assembly space limitations.

Experimental results for the unirradiated and irradiated conditions are summarized in Table 14. Simultaneous irradiation procedures were again used, with the Charpy-V specimens receiving a neutron dosage of $\sim 2 \times 10^{19}$ n/cm$^2$ (>1 Mev) at an exposure temperature less than 250°F.

Referring to the full shear energy values listed in Table 14, it is noted that the postirradiation full shear energy levels of the commercial reference heat and of the air induction heat with residuals were 32 ft-lb or less, which precluded the use of the Charpy-V 30-ft-lb criterion for the assessment of transition temperature increases of these steels. Further, the delineation of cleavage and shear zones in the fracture surfaces of the Charpy specimens of these as well as the other steels in this series was difficult to define. Thus, full crosscomparisons based on either an

TABLE 12
Heat Treatment of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats Representing the High Strength Condition

| High Strength Condition | Heat Treatment |
|---|---|
| I | Austenitized at 1650°F for 1 hour; water quenched; tempered at 1100°F for 1 hour; water quenched |
| II | Austenitized at 1650°F for 1 hour; water quenched; tempered at 1100°F for 1 hour; water quenched; stress relieved at 1050°F for 30 hours total in six 5-hour cycles |

TABLE 13
Tensile Properties of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats Representing the High Strength Condition

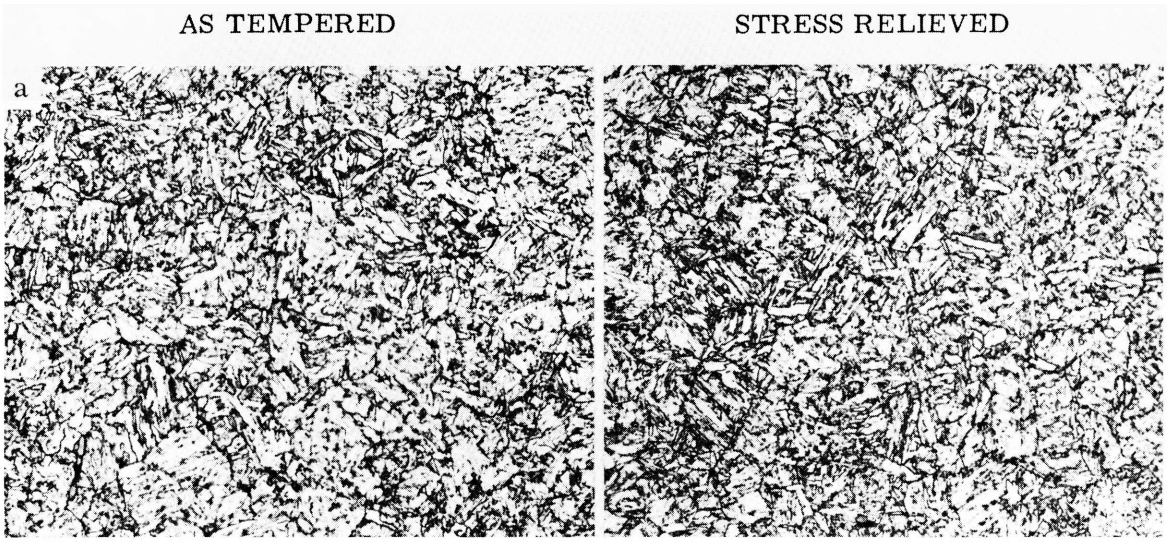
| Heat | Yield Strength (0.2% Offset)* (ksi) | | Tensile Strength† (ksi) | |
|---|---|---|---|---|
| | As Tempered | Stress Relieved | As Tempered | Stress Relieved |
| Commercial heat | 125.8 | 113.2 | 135.4 | 123.8 |
| Air induction heat with residuals | 129.9 | 114.0 | 138.1 | 122.9 |
| Air induction heat without residuals | 120.9 | 114.2 | 132.1 | 125.6 |
| Vacuum melted heat without residuals | 116.2 | 115.3 | 127.6 | 125.3 |

*0.252-in.-gage-diameter specimens.
†Average values from three specimen tests.

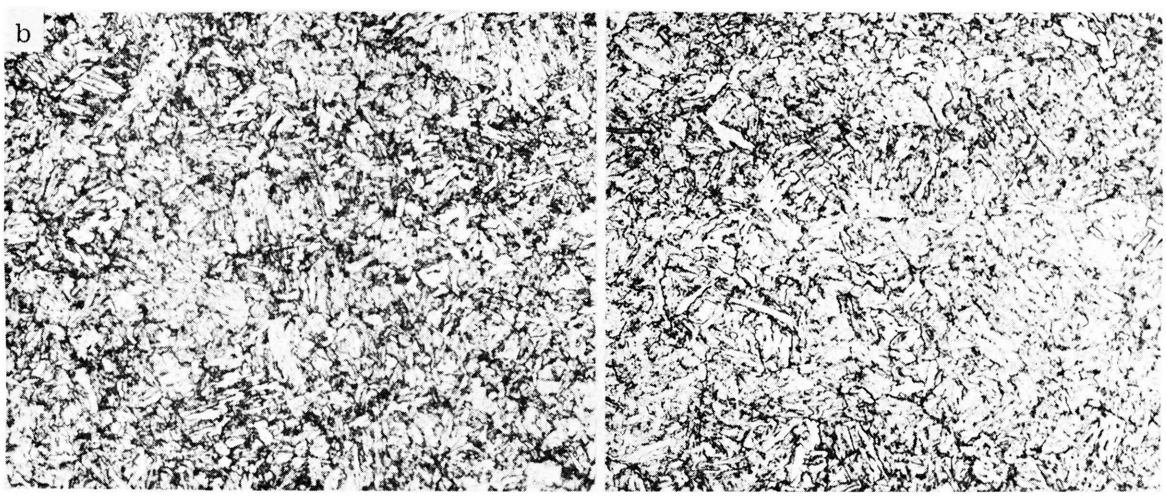
TABLE 14
Charpy-V Transition Temperature Behavior of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats in the High Strength Condition After Irradiation at <250°F to Approximately $2 \times 10^{19}$ n/cm² (>1 Mev)

| Heat | Heat Treatment Condition | Charpy-V 30-ft-lb Transition Temperature (°F) | | | Charpy-V 20-ft-lb Transition Temperature (°F) | | | Full Shear Energy Absorption (ft-lb) | |
|---|---|---|---|---|---|---|---|---|---|
| | | Initial | Irradiated | $\Delta T$ | Initial | Irradiated | $\Delta T$ | Initial | Irradiated |
| Commercial heat | Austenitized and tempered | −120 | — | — | −145 | 65 | 210 | 56 | 32 |
| | Stress relieved | − 85 | — | — | −120 | 105 | 225 | 45 | 28 |
| Air induction heat with residuals | Austenitized and tempered | − 70 | — | — | −130 | 100 | 230 | 41 | 24 |
| | Stress relieved | − 60 | — | — | −100 | 125 | 225 | 41 | 26 |
| Air induction heat without residuals | Austenitized and tempered | −120 | 15 | 135 | — | — | — | 71 | 50 |
| | Stress relieved | −125 | 0 | 125 | — | — | — | 75 | 57 |
| Vacuum melted heat without residuals | Austenitized and tempered | −170 | −90 | 80 | — | — | — | 110 | 88 |
| | Stress relieved | −125 | −30 | 90 | — | — | — | 98 | 67 |

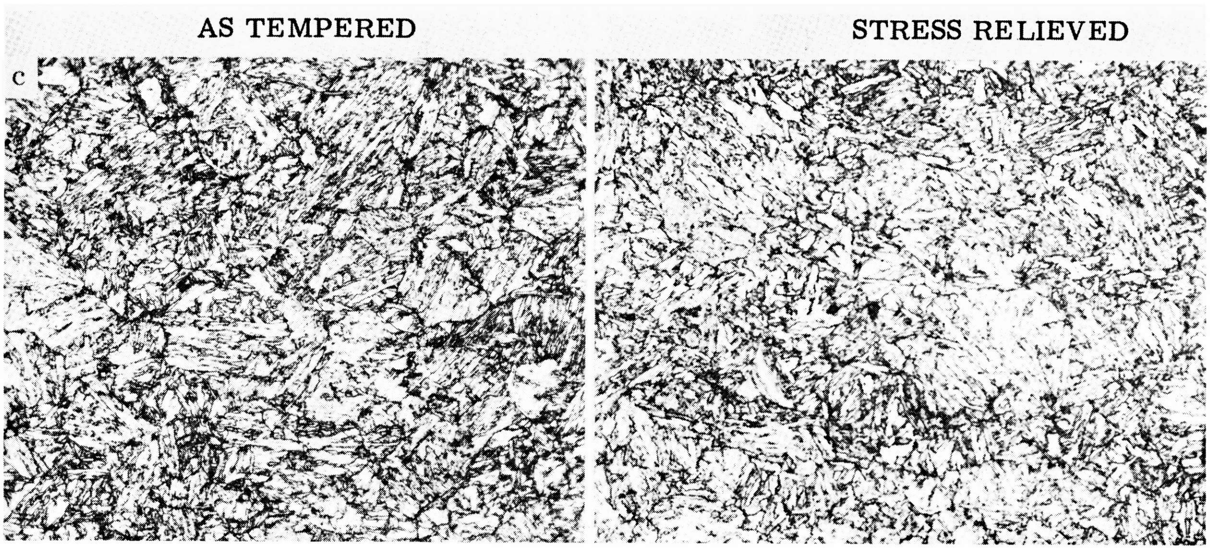
impact energy transition or 50% shear transition temperatures were not attempted. Individual assessments, however, were possible. From Charpy-V 20-ft-lb transition temperature increases, a measure of correspondence in radiation sensitivity is noted between the reference heat and the air induction heat with residuals in the as tempered condition. Similarly, the data for these heats do not signify an influence of stress relieving on irradiation response. The data for the air induction heat without residuals and the vacuum melted heat also do not indicate an effect



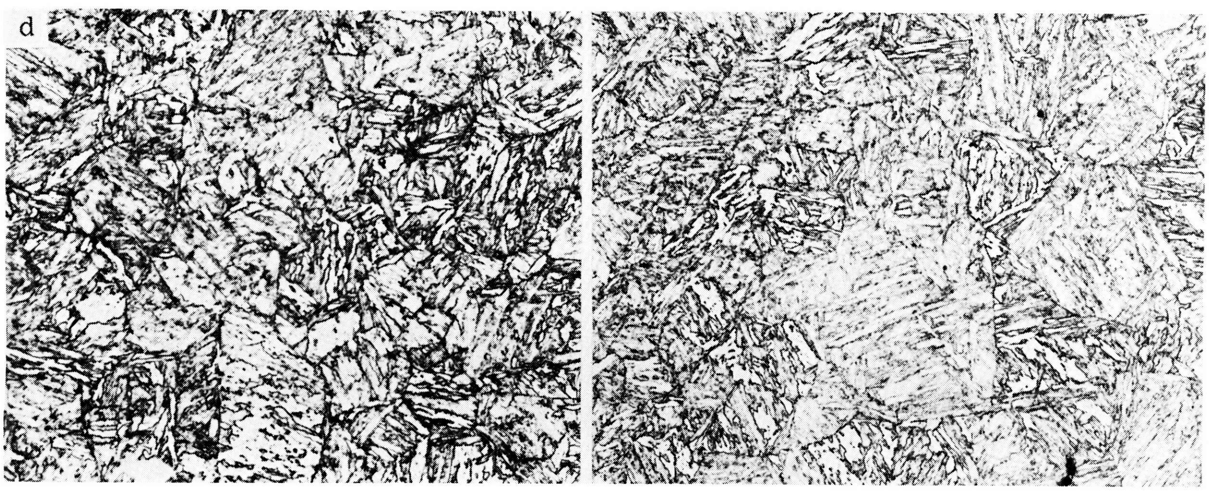

(a) Commercial heat




(b) Air induction heat with residuals




**(c) Air induction heat without residuals**




**(d) Vacuum melted heat without residuals**

Fig. 12 — Microstructures of the A302-B commercial reference heat and of the laboratory heats in the "high strength" condition. Tempered martensite (500X).

of stress relieving on radiation sensitivity. However, a difference in the respective Charpy-V 30-ft-lb transition temperature increases of these two heats is seen, from which a beneficial effect of vacuum melting, *i.e.*, a lower gas content, is shown.

On relating the general behavior patterns observed with this heat treatment series to those noted earlier with the "commercially heat treated" steels, direct correspondence is found. Specifically, experimental agreement was observed with regard to: (a) the nil-effect of a stress relieving treatment on irradiation response, (b) equivalent radiation sensitivity between the air induction heat with residuals and the commercial (open hearth) reference heat, and (c) a superior irradiation performance of the vacuum melted heat as compared to the air induction heat without residuals. In addition, the results for the two heat treatment series (Tables 9 and 14) permit a partial assessment of strength level as a factor influencing radiation sensitivity. Although the neutron dosages were not equivalent (3.0 *versus* $2.0 \times 10^{19}$ n/cm²) for both heat treatment series, the exposure difference is not considered large enough to account for the significant differences in transition temperature increase exhibited by either the air induction heat without residuals or the vacuum melted heat. Thus, it would appear that a tempered martensite structure is less radiation sensitive than a tempered upper bainite structure. This trend parallels that suggested by other research results (Fig. 2) in which the transition temperature response of the A302-B reference heat in the commercially heat treated condition (tempered upper bainite) was found to be greater than that of a commercially heat treated HY-80 steel (tempered martensite).

*Conditions 3 and 4: Low and Intermediate Strength Conditions*—The heat treatment used to achieve a "low strength" condition (condition 3 in Table 8) of the commercial reference and laboratory heats is given in Table 15. Also listed in Table 15 are the two heat treatment conditions, representing a strength level (condition 4) intermediate between the "commercially heat treated" and the "high strength" conditions, which were included in this particular irradiation series for comparison purposes. Tensile properties developed with the respective heat treatments are shown in Table 16. Microstructures of the heats are presented in Figs. 13 and 14.

Charpy-V specimens from each of the four materials in the three heat treatment conditions indicated in Table 15 were exposed simultaneously at $<250$°F to $3.2 \times 10^{19}$ n/cm² ($>1$ Mev). Experimental results are summarized in Table 17. In the case of the air induction heat without residuals, a significant degree of data scatter was observed with both the as tempered and the stress relieved conditions. The accuracy of transition temperature increases described by this particular group of specimens is considered limited to ±25°F.

Referring first to the data for the austenitized and furnace cooled condition, it is noted that the initial preirradiation transition temperatures of the heats were generally quite high, ranging from 55°F to 215°F. Commensurate with observations for the high strength steel series, the residual-free heats were again found to exhibit higher full shear energy absorption values than the residual-containing heats. On comparing the transition temperature responses to irradiation, further reinforcement of behavior trends found with the two prior steel series is observed with one significant exception. While the commercial reference heat and the air induction heat with residuals show equivalent radiation sensitivity, the air induction heat without residuals does not follow suit as might be expected from the data for the commercially heat treated condition. To the contrary, the 245°F transition temperature increase denotes an intermediate irradiation response between the behavior of the air induction heat with residuals and that of the vacuum melted heat (without residuals). Similarly, the data in Table 17 for the quenched and tempered conditions of this heat, while not permitting a precise evaluation, also are indicative of a similar relationship. Thus, a possible influence of residual elements on irradiation response is inferred by the aggregate experimental test results.

The comparison of microstructures for the low strength condition was found to further advance the suggestion that residual elements have an effect on radiation sensitivity. From Fig. 13, it is noted that the structure of the air induction heat without residuals is very similar to that of the air induction heat with residuals as well as to that of the reference heat. Thus, the lower apparent sensitivity of the air induction heat without residuals compared to the residual-containing heats cannot be ascribed to microstructural

TABLE 15
Heat Treatment of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats Representing the Low Strength Condition and the Intermediate Strength Conditions

| Condition | Heat Treatment |
|---|---|
| Low strength condition | Austenitized at 1650°F for 1 hour; furnace cooled to 1200°F; held at 1200°F for 3 hours; furnace cooled to 600°F; air cooled from 600°F |
| Intermediate strength condition I | Austenitized at 1650°F for 1 hour; water quenched; tempered at 1200°F for 1 hour, water quenched |
| Intermediate strength condition II | Austenitized at 1650°F for 1 hour; water quenched; tempered at 1200°F for 1 hour, water quenched; stress relieved at 1125°F for 30 hours total in six 5-hour cycles. |

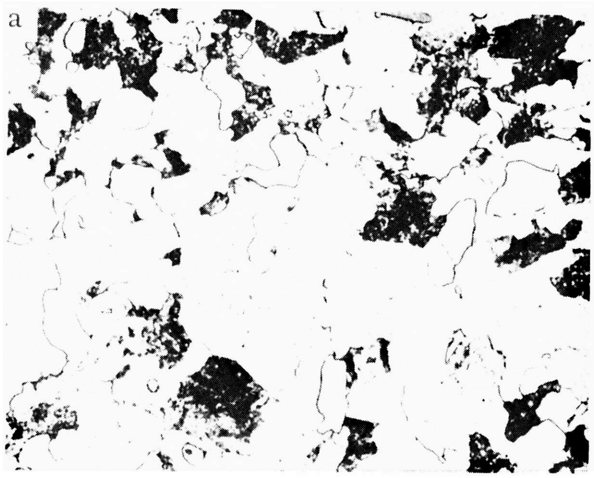
TABLE 16
Tensile Properties of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats Representing the Low and Intermediate Strength Conditions

| Heat | Yield Strength (0.2% Offset)* (ksi) | | | Tensile Strength† (ksi) | | |
|---|---|---|---|---|---|---|
| | Austenitized; Furnace Cooled (Low Strength Condition) | As Tempered (Intermediate Strength Condition I) | Stress Relieved (Intermediate Strength Condition II) | Austenitized Furnace Cooled (Low Strength Condition) | As Tempered (Intermediate Strength Condition I) | Stress Relieved (Intermediate Strength Condition II) |
| Commercial heat | 48.8 | 104.6 | 91.9 | 81.3 | 116.1 | 104.9 |
| Air induction heat with residuals | 51.1 | 116.1 | 98.2 | 85.4 | 125.5 | 108.5 |
| Air induction heat without residuals | 46.0 | 105.2 | 91.0 | 78.2 | 117.2 | 103.5 |
| Vacuum melted heat without residuals | 74.2 | 102.9 | 93.6 | 100.3 | 114.4 | 105.1 |

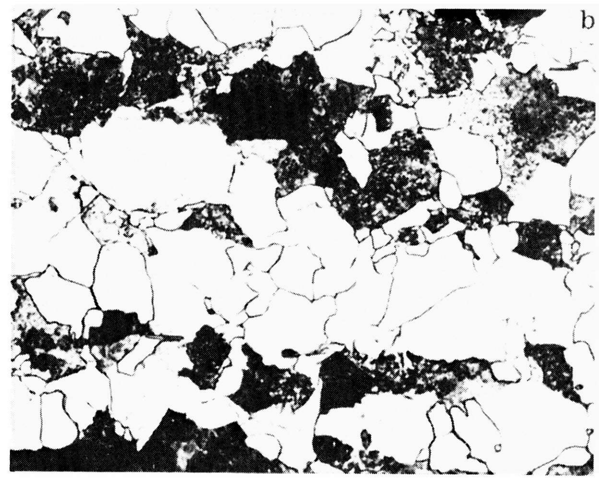
*0.252-in.-gage-diameter specimens.
†Average values from three specimen tests.

differences. With respect to the intermediate strength condition, each of the heats exhibited a tempered martensite structure as shown in Fig. 14. As with microstructural examinations of the heats in the high strength condition, some differences between individual matrix appearances are seen. If it were assumed that these differences are not significant, the data would again suggest a generally lower radiation embrittlement sensitivity with the minimization of the residual element level.

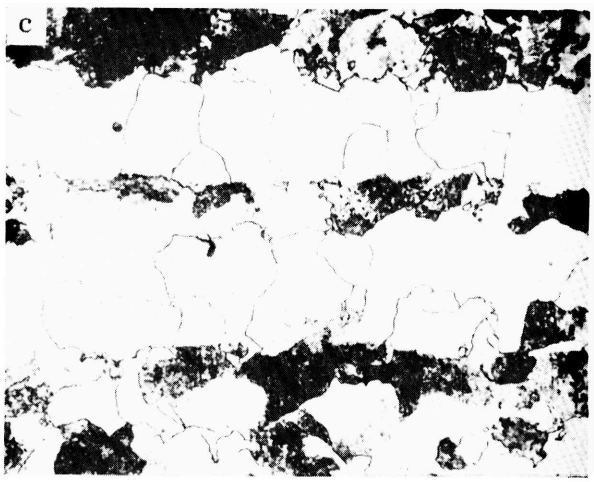
Recalling observations made with the "commercially heat treated" series (condition 1, given in Table 9), it was noted that the air induction heats with and without residuals exhibited an equivalent irradiation response in this condition. Contrary to these results, the experimental data for the low and intermediate strength conditions (conditions 3 and 4) coupled with microstructural observations indicate that residual element (P, S, Ni, Cr, Cu, Cn, *etc.*) level does play a role in radiation sensitivity development. While an apparent contradiction exists, a possible interrelationship between the influence of residual elements and microstructure is revealed from a summary evaluation of data for



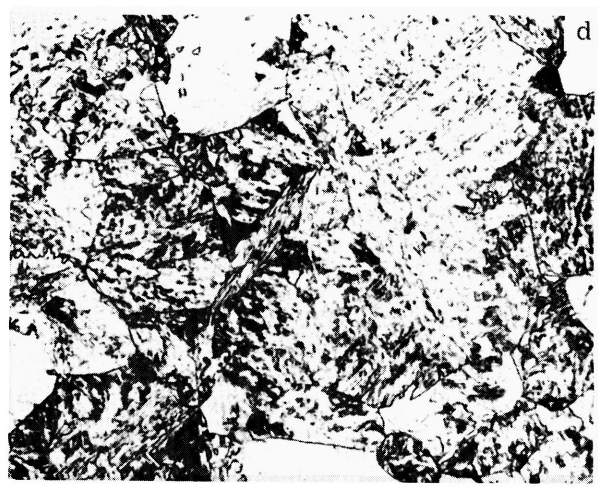

(a) Commercial heat




(b) Air induction heat with residuals




(c) Air induction heat without residuals

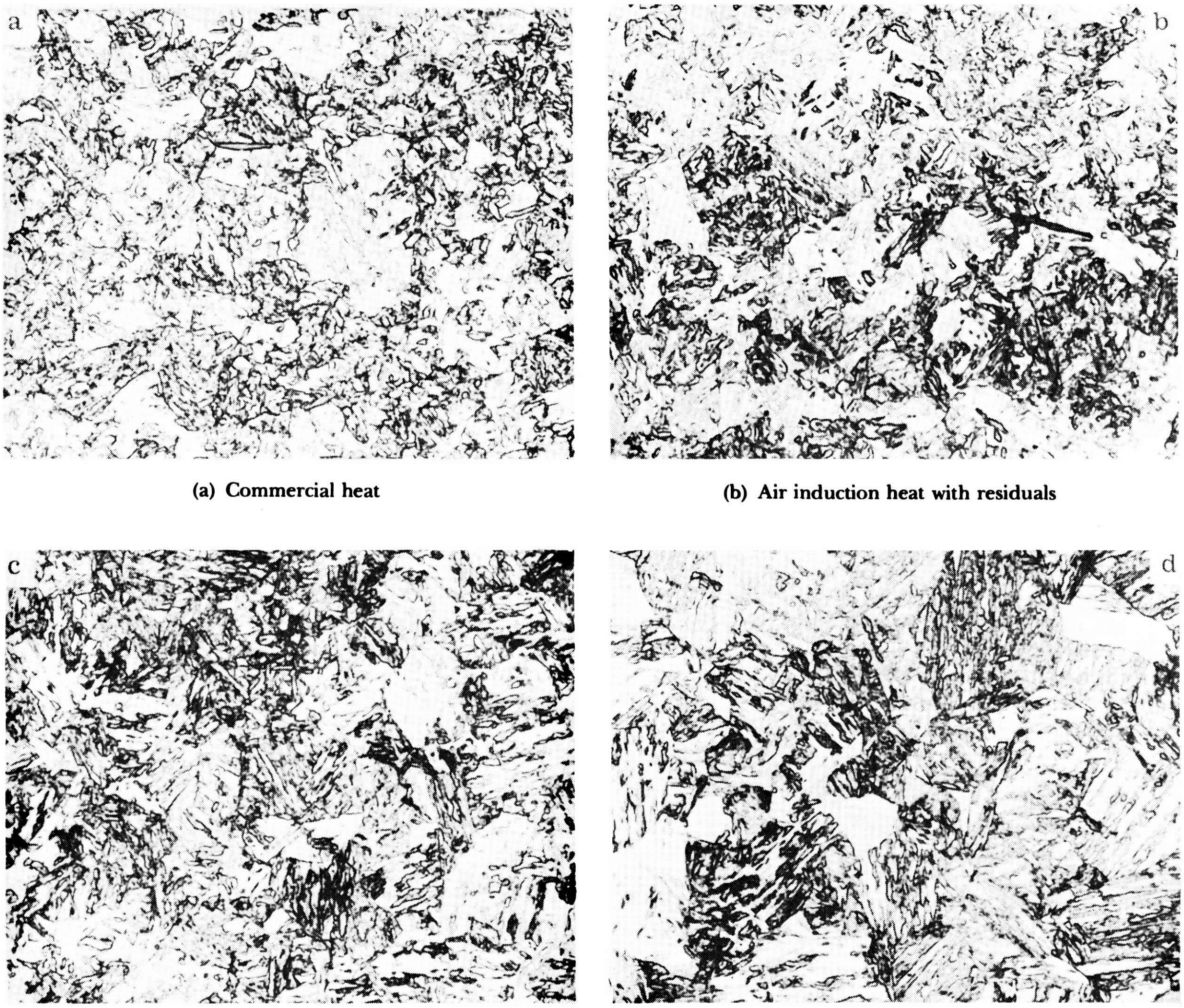



(d) Vacuum melted heat without residuals

Fig. 13 — Microstructures of the A302-B commercial reference heat and of the laboratory heats in the "low strength" condition. Upper bainite and equiaxed ferrite (500X). Pronounced banding is evident in the structures of the commercial heat and the air induction heats but not in the structure of the vacuum melted heat.

*all* heat treatment conditions. The data for the commercially heat treated condition (Table 9) and for the low strength condition (Table 17) indicate that, for tempered upper bainite or mixed upper bainite and ferrite structures, residual element level has only a secondary influence on sensitivity. On the other hand, the results for the high (Table 14) and intermediate strength conditions (Table 17) illustrate that for tempered martensite structures, residual levels have a marked effect on irradiation response. A more complete investigation of the dependency of residual element effects on microstructure has been undertaken.

The equivalence of neutron exposures for the irradiations of the heats in the low and intermediate strength conditions and in the commercially heat treated condition (3.2 *versus* $3.1 \times 10^{19}$ n/cm² > 1 Mev) permits one further correlation of irradiation behavior to microstructure. Examining the experimental results presented in Tables 9 and 17, very similar increases in transition temperature are found between the low strength and




(a) Commercial heat

(b) Air induction heat with residuals

(c) Air induction heat without residuals

(d) Vacuum melted heat without residuals

Fig. 14 — Microstructures of the A302-B commercial reference heat and of the laboratory heats in the "intermediate strength" as tempered condition. Tempered martensite (500X). Cyclic stress relief annealing treatments were not found to appreciably alter these structures.

commercially heat treated conditions. With reference to the air induction heats and the commercial heat, this agreement would signify an equivalent irradiation response for tempered upper bainite and mixed upper bainite-ferrite structures. In contrast to this level of response, the tempered martensite structures of the intermediate strength condition depict a generally lower radiation embrittlement sensitivity, confirming the analysis presented with the results of the high strength condition above.

## Program 3: Evaluation of Nickel Content as a Radiation Sensitivity Variable

The influence of nickel as an alloying addition on radiation sensitivity was assessed through the irradiation of five Ni-Cr-Mo steels from selected 300-lb laboratory heats. As shown in Table 18, the steels were closely matched in chemistry with the exception of nickel content, which ranged from 3.8 to 8.4 wt-%. Since some variation in hardenability and continuous cooling transformation

TABLE 17

Charpy-V Transition Temperature Behavior of the A302-B Commercial Reference Heat and of the 300-lb Laboratory Heats in the Low and Intermediate Strength Conditions (see Table 15) After Irradiation at <250°F to $3.2 \times 10^{19}$ n/cm² (>1 Mev)

| Materials | Condition | Charpy-V 30-ft-lb Transition Temperature (°F) | | | Full Shear Energy Absorption (ft-lb) | |
|---|---|---|---|---|---|---|
| | | Initial | Irradiated | $\Delta T$ | Initial | Irradiated |
| Commercial heat | Austenitized; furnace cooled | 75 | 395 | 320 | 65 | 43 |
| | As tempered | −120 | 170 | 290 | 55 | 45 |
| | Stress relieved | −105 | 195 | 300 | 64 | 44 |
| Air induction heat with residuals | Austenitized; furnace cooled | 120 (95*) | 430 (425*) | 310 (330*) | 49 | 42 |
| | As tempered | − 90 (−110*) | − (205*) | − (315*) | 45 | 30 |
| | Stress relieved | − 75 (−90*) | 230 (215*) | 305 (305*) | 51 | 33 |
| Air induction heat without residuals | Austenitized; furnace cooled | 55 | 300 | 245 | 87 | 87 |
| | As tempered | −175 | 5† | 180† | 98 | 82 |
| | Stress relieved | −135 | −5† | 130† | 93 | 83 |
| Vacuum melted heat without residuals | Austenitized; furnace cooled | 215 | 390 | 175 | 84 | 72 |
| | As tempered | −185 | −50 | 135 | 112 | 95 |
| | Stress relieved | −135 | 10 | 145 | 111 | 101 |

*Charpy-V 25-ft-lb transition temperature.
†Irradiated specimens exhibited somewhat high data scatter.

characteristics would be expected with this range of nickel additions, the steels in 1/2-in.-plate form were placed in the quenched and tempered condition to achieve a similarity of microstructures. The heat treatment employed included austenitizing at 1500°F for 1 hour, water quenching, tempering at 1150°F for 2 hours, and water quenching. The structure developed in each case was tempered martensite and/or lower bainite as illustrated in Fig. 15. As noted from Table 18, an equivalence in tensile properties of the steels was achieved with one exception, the 7.3-wt-%-nickel steel. The yield and tensile strengths of this steel (128.5 and 144.8 ksi respectively), were somewhat lower than the 143 to 150 ksi yield strengths and the 153 to 162 ksi tensile strengths developed in the remaining steels.

Charpy-V specimens of all steels were irradiated simultaneously at <250°F to $1.2 \times 10^{19}$ n/cm² (>1 Mev), while the 0.252-in.-diameter tension test specimens were exposed at <250°F to $1.5 \times 10^{19}$ n/cm². The experimental results are presented in Tables 19 and 20. On examining these data, no consistent behavior pattern or trend indicative of a relationship between nickel content and radiation sensitivity is found. The small variations in irradiation response observed may be related to slight differences in the microstructures of the respective steels or possibly to small differences in the gas contents.

The results of this experiment show conclusively that nickel content within the range investigated does not exert a direct influence on radiation sensitivity. However, microstructure has been shown to be a primary factor in radiation sensitivity level; thus an indirect influence of nickel content through modification of

TABLE 18
Tensile Strength and Chemical Composition of Five Ni-Cr-Mo Steels Varying in Nickel Content From 3.8 to 8.4 wt-%

| Steel | Nickel (wt-%) | Yield Strength (0.2% offset) (ksi) | Tensile Strength (ksi) | Chemical Analysis (wt-%) | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | C | Mn | P | S | Si | Ni | Cr | Mo |
| A | 3.8 | 149.4 | 158.5 | 0.17 | 0.23 | 0.009 | 0.006 | 0.23 | 3.8 | 0.87 | 1.04 |
| B | 4.9 | 143.9 | 153.5 | 0.14 | 0.23 | 0.010 | 0.005 | 0.22 | 4.9 | 0.87 | 1.01 |
| C | 6.4 | 146.0 | 158.5 | 0.17 | 0.24 | 0.008 | 0.005 | 0.24 | 6.4 | 0.89 | 1.06 |
| D | 7.3 | 128.5 | 144.8 | 0.14 | 0.23 | 0.014 | 0.005 | 0.25 | 7.3 | 0.89 | 1.06 |
| E | 8.4 | 148.3 | 161.9 | 0.16 | 0.23 | 0.008 | 0.004 | 0.23 | 8.4 | 0.89 | 1.02 |

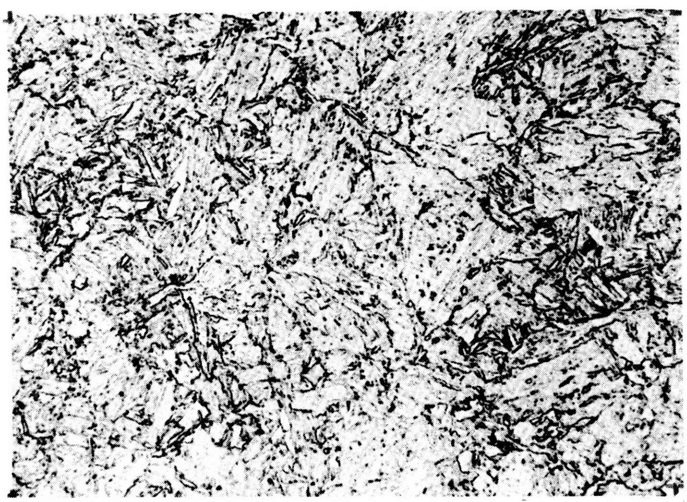


Fig. 15 — Typical microstructure of Ni-Cr-Mo steels varying in nickel content from 3.8 to 8.4 wt-%. Tempered martensite and/or lower bainite (500X).

TABLE 19
Charpy V-Notch Ductility Characteristics of Five Ni-Cr-Mo Steels Varying in Nickel Content From 3.8 to 8.4 wt-% Before and After Irradiation at <250°F to $1.2 \times 10^{19}$ n/cm² (>1 Mev)

| Steel | Nickel (wt-%) | 30-ft-lb Transition Temperature (°F) | | | Full Shear Energy Absorption (ft-lb) | | |
|---|---|---|---|---|---|---|---|
| | | Initial | Irradiated | $\Delta T$ | Initial | Irradiated | $\Delta$ft-lb |
| A | 3.8 | −125 | 70 | 195 | 71 | 35 | 36 |
| B | 4.9 | −125 | 80 | 205 | 54 | 34 | 20 |
| C | 6.4 | −205 | − 40 | 165 | 48 | 37 | 11 |
| D | 7.3 | −295 | − 80 | 215 | 56 | 37 | 19 |
| E | 8.4 | −300 | −150 | 150 | 55 | 36 | 19 |

TABLE 20
Tensile Properties of Five Ni-Cr-Mo Steels Varying in Nickel Content From 3.8 to 8.4 wt-% Before and After Irradiation at <250°F to 1.5 × $10^{19}$ n/cm² (>1 Mev)

| Steel | Nickel (wt-%) | Yield Strength (ksi) | | | Tensile Strength (ksi) | | | Elongation (%) | | Reduction of Area (%) | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Initial | Irrad. | Δksi | Initial | Irrad. | Δksi | Initial | Irrad. | Initial | Irrad. |
| A | 3.8 | 149.4 | 180.4 | 31.0 | 158.5 | 184.0 | 25.5 | 19.0 | 14.1 | 66.0 | 61.7 |
| B | 4.9 | 143.9 | 173.5 | 29.6 | 153.5 | 176.3 | 22.8 | 19.0 | 12.7 | 62.5 | 55.7 |
| C | 6.4 | 146.0 | 172.9 | 26.9 | 158.5 | 175.2 | 16.7 | 19.6 | 13.5 | 62.9 | 60.0 |
| D | 7.3 | 128.5 | 160.0 | 31.5 | 144.8 | 163.9 | 19.1 | 20.9 | 15.6 | 63.8 | 60.2 |
| E | 8.4 | 148.3 | 175.8 | 27.5 | 161.9 | 182.4 | 20.5 | 20.4 | 15.9 | 66.0 | 61.5 |

continuous cooling transformation characteristics must be assumed to exist.

## SUMMARY AND CONCLUSIONS

Experimental evaluations of possible factors causing variability in radiation embrittlement sensitivity demonstrate that the ultimate goals of isolating the critical metallurgical variables and, further, the application of this knowledge to the development of advanced steels exhibiting consistently low embrittlement sensitivity are realistic and attainable. However, the full assessment of the singular and combined influences of the many suspect variables will require a large concerted experimental effort. The exploratory studies reported herein, for example, are but a part of a comprehensive series of investigations undertaken jointly by NRL and the U.S. Steel Corporation, Applied Research Laboratory. The significant research findings in the isolation and assessment of dominant influences in radiation sensitivity development may be enumerated as follows:

1. The radiation embrittlement sensitivity of a steel can be altered appreciably through heat treatment practices.
2. A complete reversal in the order of radiation embrittlement sensitivity of two steels, HY-80 and A350-LF3, upon which the limits of the NRL trend band for <450°F irradiations were primarily based, was possible through reheat treatment procedures.
3. Microstructure plays a dominant, if not the most influential, role in radiation sensitivity development.
4. A tempered martensite structure is generally less radiation sensitive than tempered upper bainite or ferrite structures.
5. Vacuum melting and the minimization of residual element content yields steels which exhibit lower radiation embrittlement sensitivity than steels produced by conventional open hearth melting, regardless of the final heat treatment condition.
6. A302-B steels produced by conventional open hearth and air induction melting practices do not appear to differ in their degree of radiation embrittlement for the case of matching residual contents.
7. Stress relieving heat treatments do not alter the irradiation response of A302-B steel even if initially producing an increase in the Charpy-V transition temperature and a small reduction in yield and tensile strengths.
8. Radiation embrittlement sensitivity is not influenced directly by nickel content in the range 3.8 to 8.4 wt-%. However, an indirect influence of nickel content, through modification of hardenability and continuous cooling transformation characteristics, is quite plausible.

In addition to item 5 above, assessments of the irradiation response of an air induction heat with residuals *versus* an air induction heat without residuals are suggestive of some contribution of residual element level to radiation embrittlement sensitivity. A more definitive evaluation of this total contribution as well as the examination of the separate and combined influences of carbon, phosphorus, and sulfur content are a few of the objectives of the continuing experimental program. Similarly, the individual effects of oxygen and nitrogen levels on irradiation response are being investigated in view of the superior performance of the vacuum melted A302-B heat.

## THE CONTINUING EXPERIMENTAL PROGRAM

Although basic trends in radiation sensitivity as a function of metallurgical variables are becoming apparent, the testing of these trends with elevated temperature irradiations (>500°F) is considered necessary to arrive at a more complete understanding of radiation embrittlement development. The performance of irradiation experiments having this objective is included in the current experimental program. Significant research developments from these and other associated investigations will be presented in future topical reports and publications.

## ACKNOWLEDGMENTS

The NRL Metallurgy Division research on critical factors responsible for variable radiation embrittlement sensitivity of steels is supported jointly by the Office of Naval Research, the U.S. Atomic Energy Commission, Division of Reactor Development and Technology, and the Army Nuclear Power Program.

The authors gratefully acknowledge the efforts of the U.S. Steel Corporation, Applied Research Laboratory, in providing the special laboratory heats required for this research program as well as their assistance in the reheat treatment of selected commercially produced steels. The authors particularly thank Mr. James V. Alger of the Applied Research Laboratory for his personal interest and active participation in the planning of these investigations. The success of the recent experimental effort in isolating several of the critical metallurgical variables controlling radiation sensitivity are measurably attributed to his may ideas and helpful suggestions as to the experimental approach and to his direction of the melting and heat treatment of the special steels used in this program. The continuing interest and cooperation of Mr. M. W. Lightner, Vice President, U.S. Steel Corporation, Research and Technology Division, in supplying requisite materials for the NRL irradiation effects studies are also greatly appreciated. Further, the authors thank the Physics Group of the MTR Staff of the Idaho Nuclear Corporation for their valuable assistance in the neutron dosimetry analyses. The personal efforts and contributions of each member of the staff of the Reactor Materials Branch toward the accumulation of the irradiation effects data are also acknowledged with gratitude.

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3. REPORT TITLE

INITIAL EVALUATIONS OF METALLURGICAL VARIABLES AS POSSIBLE FACTORS CONTROLLING THE RADIATION SENSITIVITY OF STRUCTURAL STEELS

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

An initial report on one phase of the problem.

5. AUTHOR(S) *(Last name, first name, initial)*

Hawthorne, J.R., and Steele, L.E.

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|---|---|---|
| September 29, 1966 | 36 | 15 |

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|---|---|
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13. ABSTRACT


Experimental investigations for the isolation and assessment of metallurgical factors causing variable radiation embrittlement sensitivity of reactor structural steels have been undertaken, using both large-tonnage commercial heats and special laboratory heats of steel. Metallurgical variables being evaluated include the identity and quantity of major alloying elements and of residual elements, steel-making practice—both melting (refining) and heat treatment practice, microstructure, and gas content.

Experimental results from the initial series of the exploratory screening studies demonstrate that the radiation sensitivity of a steel can be altered appreciably through heat treatment practices and that microstructure plays a dominant, if not the most influential, role in radiation sensitivity development. A tempered martensite structure was noted to be generally less radiation sensitive than tempered upper bainite and ferrite structures. The data also indicate that vacuum melting and the minimization of residual element content yields steels having a superior irradiation performance compared with steels produced by conventional open hearth melting. However, long-term stress relieving heat treatments were not found to alter the irradiation response of A302-B steel.


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