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RECENT DATA ON THE EFFECTS OF NEUTRON IRRADIATION ON STRUCTURAL METALS AND ALLOYS

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Introduction

A number of papers have been published in the past two years on the effects of neutron irradiation on structural metals. Among the many interesting observations reported in these papers, the most outstanding are probably the increased strengths, the increased brittleness, and the change in mechanical test behavior of a number of metals and alloys after irradiation. This paper presents some recent observations of such property changes in irradiated metals. Although the data now available on radiation effects in metals are not sufficiently complete to permit them to be used either in design calculations or in the formulation of a rigorous theory of radiation effects, the results presented here and in other papers should serve both to caution the reactor designer and to suggest profitable studies by other investigators.

Data will be presented from tensile and notched-bar impact tests on several carbon steels, a carbon steel weldment, a nickel steel, a high-strength steel, and a high-purity iron.

Several papers[1,2] have presented theories of neutron effects in metals, and the subject will not be reviewed in this paper. The changes in properties reported in this paper are thought to be due almost exclusively to bombardment by energetic neutrons, and, as a convention, integrated neutron fluxes (doses) reported in this paper are for neutrons of energies greater than 1 Mev. The measurements of fast neutron fluxes and doses is difficult, and therefore many of the values reported here and elsewhere are estimates.

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1. F. Seitz, Discussions Faraday Soc. 5, 271 (1949).
2. D. S. Billington, Nucleonics (9), 14, 54 (1956).

The techniques and difficulties of irradiation studies have been discussed in the literature[3,4] and will be reported only briefly. The scarcity of data from neutron irradiations at elevated temperatures is not due to a lack of interest but must be attributed to the difficulty and expense of such investigations. The greater part of the data presented here is for irradiation temperatures of less than 200°F. Some data will be presented on elevated temperature irradiations to low neutron doses. Such data for higher neutron doses should be available in the relatively near future.

Specimens

The specimens machined from the steels investigated were of substandard size so that a greater number of specimens could be irradiated in the available exposure facilities. The tensile test specimens were of 0.180-in.-gage diameter and 1-in.-gage length with 1/4-in. square or round ends for gripping in the tensile machine. The subsize notched-bar impact specimens were multiple notch Izod type, 0.2 in. square with a notch 0.060 in. deep, an included angle of 45 degrees, and a root radius of 0.005 in. Notch to striker distance was 7/8 in. All specimens were machined with their longitudinal axis parallel to the rolling direction of the plate and with the notches in the impact specimens perpendicular to the plate surface.

Irradiation Conditions

Specimens were irradiated either in a horizontal beam hole facility (HB-3) of the Low Intensity Test Reactor (LITR)[5] at Oak Ridge or in a horizontal beam hole facility (HB-3) of the Materials Testing Reactor (MTR)[5] at Arco, Idaho.

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3. R. G. Berggren, S. E. Dismuke, M. J. Feldman, and J. C. Wilson, "The Mechanics of Testing Irradiated Materials," presented at the 2nd Pacific Area Meeting ASTM, Sept. 17-21, 1956, Los Angeles, Calif. (To be published).
4. "Fourth Annual Symposium on Hot Laboratories and Equipment," TID-5280 (Sept. 1955) and Supplement 1 (Jan. 1956).
5. "Research Reactors," TID-5275.

All specimens not purposely held at elevated temperatures were cooled by contact with water-cooled metal walls. The temperatures of these specimens did not exceed 200°F during irradiation. Specimens held at elevated temperatures during irradiation were mounted in miniature furnaces, and temperatures were monitored with thermocouples welded to the specimens.

Integrated fast neutron fluxes for irradiations in the LITR and MTR were determined either by measurement of sulfur monitors included in the experiment and irradiated simultaneously with the test specimens or were calculated from such data obtained during a subsequent experiment and corrected for reactor operating conditions. The maximum dose reached in LITR irradiations was 5×10^{18} nvt and in the MTR irradiations was 1.3×10^{20} nvt.

Test Procedure

All tensile specimens, both irradiated and unirradiated, were tested at room temperature in a vertical, screw-loaded, tensile tester[6] with a "hard," electronic, load-measuring system. The specimens were pulled at a strain rate of 0.05 $min.^{-1}$ except where otherwise noted. Elongation measurements are accurate within ±1% (not 1% of reading).

The impact specimens were tested in a modified Tinius Olsen plastics impact tester[7] adapted for remote testing of subsize metal specimens. Test specimens and vise were brought to test temperature in position for test. Test temperatures were determined by means of a thermocouple attached to the vise and adjacent to the specimen.

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6. J. C. Wilson, R. G. Berggren, W. W. Davis, and R. A. Hall, p 43, "Fourth Annual Symposium on Hot Laboratories and Equipment," TID-5280 Supplement (Jan. 1956).
7. W. Pate et al., Nucleonics 10 (6), 60 (1952). The machine has been considerably altered since this article was written.

Materials

The metals used in this series of irradiation studies are described below. The mechanical test values given in this section were provided by the supplier. Chemical analyses were made by either the supplier or the author's laboratory. The item numbers are the author's identification of each individual lot of material.

High-Purity Iron

Item 25 is a vacuum-melted and rolled iron obtained in the form of $\frac{1}{4}$-in. square rod. Chemical composition was 0.004% C, 0.0038% O_2, 0.000073% N_2, 0.0028% Ni, and 0.009% Si; other metallic impurities were less than 0.001% each. Specimens were annealed at 1150°F (below the critical temperature) for $\frac{1}{2}$ hr and water quenched.

8½% Nickel Steel

Item 42 is a high-nickel steel manufactured to ASTM Specification A-353-53T and was obtained in the form of 3/8-in. plate. Heat treatment, by the supplier, consisted in double normalizing from 1650 and 1450°F and reheating at 1050°F.[8] Chemical composition was 0.10% C, 0.76% Mn, 0.23% Si, and 8.58% Ni. Grain size was smaller than ASTM-8, and ASTM inclusion rating was A-1 thin.

Hot-Rolled Carbon-Silicon Steel

Item 43 is a carbon-silicon steel manufactured to ASTM Specification A-212-52aT, Grade B, in the form of hot-rolled, 5/8-in. plate. Chemical composition was 0.20% C, 0.82% Mn, 0.018% P, 0.030% S, and 0.22% Si. Grain size was ASTM-7,

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8. G. R. Brophy and A. J. Miller, Trans. ASM 41, 1185 (1949).

and ASTM inclusion rating was C-2 heavy. Mechanical test values on standard specimens were 46,700 psi yield point; 79,600 and 78,400 psi tensile strength; and 26% elongation in 8 in. Homogeneity and bending tests were satisfactory. Specimens machined from this steel were given no further heat treatment.

Hot-Rolled Carbon Steel

Item 44 is a carbon-steel manufactured to ASTM Specification A-285-52aT, Grade A, as hot-rolled, 3/4-in. plate. Chemical composition was 0.11% C, 0.36% Mn, 0.018% P, and 0.02% S. Grain size was ASTM-7, and inclusion rating was A-3 thin. Mechanical test values on standard specimens were 32,400 psi yield point; 51,700 and 49,600 psi tensile strength; and 34% elongation in 8 in. Homogeneity and bending tests were satisfactory. Specimens from this steel were left in the hot-rolled condition.

Chromium-Molybdenum Steel

Item 45 is a chromium-molybdenum pressure vessel steel manufactured to ASTM Specification A-301-52aT, Grade B, as 3/4-in. plate. Chemical composition was 0.11% C, 0.36% Mn, 0.02% P, 0.026% S, 0.22% Si, 0.98% Cr, and 0.47% Mo. Grain size was ASTM-5, and inclusion rating was A-1 thin. Mechanical test values on standard specimens (normalized at 1650 to 1700°F for 1 1/2 hr, furnace cooled to 1100°F, and air cooled) were 40,600 yield point; 65,700 and 64,600 psi tensile strength; and 29% elongation in 8 in. Homogeneity and bending tests were satisfactory. Specimens used in the irradiation studies were normalized from 1700°F.

Seamless Carbon-Steel Pipe

Item 55 is a carbon-steel pipe for high-temperature service manufactured to ASTM Specification A-106-52T in the form of heavy wall pipe. This was a relatively coarse-grained steel for comparison tests with Item 56, a fine-grain, aluminum-killed

steel. Chemical composition was 0.24% C, 0.72% Mn, 0.018% P, 0.023% S, and 0.20% Si. Grain size was ASTM-2.

Item 56 is almost identical to Item 55 except that it was aluminum-killed to secure a fine-grain structure. Chemical composition of Item 56 was 0.24% C, 0.70% Mn, 0.019% P, 0.020% S, 0.19% Si; and aluminum per fine-grain practice. Grain size was ASTM-7. The chemical compositions of these two steels are almost identical except for the aluminum addition in the fine-grain steel.

Bars cut from both the above steels were normalized at 1700°F for 1 hr and air cooled before machining of test specimens.

High-Yield-Strength Alloy Steel

Item 60 is a high-yield-strength alloy steel manufactured under the trade name "Carilloy T-1"[9,10] and was obtained as quenched and tempered 1/2-in. plate. Chemical analysis and other data on this steel are not yet available.

Welded Carbon-Silicon Steel Plate

Item 65 is a weldment in a stainless-steel-clad carbon-silicon steel plate. The design of this weldment is shown in Fig. 1. The carbon steel base plate conformed to ASME Boiler Code Specification SA 212, Grade B, firebox quality and was manufactured to SA 300, aluminum-killed, fine-grained low-temperature practice. The stainless steel cladding was not included in this series of tests. The portion of the weld joint from which specimens were taken consisted of multiple passes with

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9. L. C. Bibber et al., Trans. ASME, 74, 269 (1952).
10. United States Steel Corp., "USS Carilloy T-1 Steel-Technical Data."

a low-hydrogen, carbon-steel, coated electrode, E7016. All specimens were cut from this plate with longitudinal axes perpendicular to the weld and parallel to the plate face. Notches of all the impact specimens were perpendicular to the plate face. Tensile and impact specimens referred to as "base plate" were from portions of the SA 212 plate sufficiently remote from the weld to be unaffected by the welding process. The test sections of impact and tensile specimens referred to as "weld" consisted entirely of deposited weld metal and were taken such that no dilution by the deposited iron passes had occurred. The impact specimens referred to as "heat affected zone" were made such that the notch (and subsequent test fracture) was in the heat affected zone of the base plate (within 1/16 in. of the fusion line). Specimens were given no subsequent heat treatment. Chemical analyses and other information on this weldment will be available at a later date.

Results and Discussion

Impact Tests

The results of notched-bar impact tests on these steels are shown in Figs. 2 through 10. The behavior of the two steels in Figs. 2 and 3 at about 5×10^{18} nvt is typical of many carbon steels at this neutron dose. The higher ductile-to-brittle transition temperature and lower fracture energy of the ductile (high temperature) part of the curve after irradiation are common features of such curves. The magnitude and the characteristics of these changes will vary from one steel to another, depending upon a number of factors.

The effects of increasing neutron dose (and also neutron flux) are shown in Fig. 4. The increases in fracture transition temperature and decrease in fracture energy in the "ductile" part of the curve are more evident at the higher neutron dose. This figure does not indicate any saturation of radiation

effects. Within the accuracy of the flux and fracture energy measurements it is possible that the effects observed are roughly proportional to the neutron dose. This does not appear to be true for other steels, as can be seen in Figs. 5 and 10.

The notched-bar impact behavior of an irradiated high-tensile-strength alloy steel (Item 60) is shown in Fig. 5. The increasing brittleness at progressively greater neutron doses is very clear. In addition, the effect of irradiation at higher temperatures is depicted. The effects of irradiation at 600°F is not so great as during the same irradiation at less than 200°F.

Figure 6 gives the results of notched-bar impact tests on an 8 1/2% nickel steel. This steel, having excellent low-temperature notch ductility before irradiation, also suffers degradation of properties upon irradiation.

The effect of grain size upon the sensitivity of a steel to neutron irradiation was studied by irradiation of two similar steels (Items 55 and 56), one of which was aluminum killed to secure a fine-grain size. The results of these tests are given in Figs. 7 and 8. The unirradiated fine-grain steel showed a lower ductile-brittle transition temperature and higher "ductile" energy than did the unirradiated coarse-grain steel. This relationship was also true after irradiation to about 10^{20} nvt. The transition temperature of the irradiated coarse-grain steel was about 300°F, and that of the irradiated fine-grain steel was less than 200°F. The coarse-grain steel showed a slightly greater radiation-induced increase in transition temperature than did the fine-grain steel. These results agree with ductility results from tension tests to be discussed later. Although these results seem to indicate some superiority of the fine-grain steel, such a conslusion must be tentative in that only two heats of steel were studied.

The results of impact tests of another irradiated fine-grain steel (Item 65, base plate) are shown in Fig. 9. In this steel, irradiation at 600°F to a dose of about 5×10^{18} nvt resulted in impact properties almost identical to those of the unirradiated steel. Irradiation at less than 200°F again resulted in substantial changes in impact properties.

Results of impact tests on a weld metal (Item 65 - Weld) and the heat-affected zone (Item 65 - Heat Affected Zone) near the weld are given in Fig. 10. The impact properties as measured by transition temperature and "ductile" energy of both the deposited weld and the heat-affected zone were superior to those of the base plate (Fig. 9) before irradiation. Both the irradiated weld metal and the heat-affected zone showed the greatest changes in impact behavior that have been observed in this study. However, comparison of Figs. 9 and 10 will show that the impact characteristics of the weld metal still appear to be better than those of either the base plate or heat-affected zone at the highest neutron doses.

Tensile Tests

The results from tensile tests on several steels and on iron are tabulated in Table 1 through 4. Several unusual features may be observed at neutron dosages of 10^{19} to 10^{20} nvt. In the more-radiation-sensitive steels, high dosages reduce the work-hardening capabilities to low values, Figs. 11 and 12. As a result the yield stress and the ultimate tensile strength of these steels are practically identical, and about 2% (or less) uniform elongation precedes necking. All steels tested did show elongation during necking.[11] The postnecking elongation appears to be dose sensitive but to a lesser degree than is the uniform elongation.

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11. Some important measurements, that is, reduction of area, have not yet been made and all specimens in the test series have not been broken.

The results of tensile tests on the two alloy steels are shown in Table 1. Both steels had approximately the same yield and ultimate strengths after 1×10^{20} nvt dose but the 8 1/2% nickel steel had a higher uniform elongation. This relationship was also shown by the load-elongation curves, the 8 1/2% nickel steel showing a region of some work hardening (similar to the uppermost curve of Fig. 11), while the high-yield-strength alloy steel showed practically no work hardening (similar to the uppermost curves of Fig. 12).

The fine-grain steel appears to be less sensitive to radiation than does the coarse-grain steel (Table 2), although the load-elongation curves for both steels showed a region of some work hardening. These results are in substantial agreement with the notched-bar impact test results, and, tentatively, a fine-grain size seems to be a necessary, though not sufficient, condition for radiation resistance.

In high-purity iron the shape of the load-elongation curve was markedly affected by an increase in strain rate by a factor of 40. At a strain rate of 0.05 min^{-1} the irradiated iron showed a reduced uniform (but not postnecking) elongation, but the load-elongation curve was unusual in that the yield strength exceeded the conventional ultimate tensile strength. At a strain rate of 2.0 min^{-1} the irradiated iron showed no observable uniform elongation; virtually all the elongation occurred during necking. Postnecking elongation was not greatly affected by the irradiation. The shape of the load-elongation curve at the higher strain rate was similar to the uppermost curve of Fig. 12.

Tensile tests on the irradiated carbon steel weldment (Item 65) yielded the results shown in Table 4 and in Figs. 11 and 12. The carbon-silicon steel-base plate showed no unexpected increases in yield and tensile strengths and reductions in uniform and total elongations. The load-elongation curves, Fig. 11, were

conventional in that the steel retained some work-hardening capabilities even at the highest dose. The deposited weld metal, however, showed no observable work-hardening capability at 1.7×10^{19} nvt, and higher and local deformation (necking) occurred immediately after initial yielding, Fig. 12. The one tensile test of weld metal irradiated at an elevated temperature, Table 4, tentatively indicates that an elevated temperature lessens the effects of irradiation. These data are not conclusive in that they are for a relatively low neutron dose. Such data for higher neutron doses will be available at a later date.

Conclusions

It has been shown that neutron irradiation, in addition to increasing the yield and tensile strengths and reducing the ductility of structural steels, can also change the behavior of those steels to such an extent that they become unfamiliar materials for structural components such as pressure vessels. The designer of a nuclear power plant may be faced with the possibility of brittle fracture of components at temperatures higher than previously assumed. The possibility that these deleterious effects will be lessened at the higher irradiation temperatures expected in nuclear power plants appears to be good. Further irradiation studies at elevated temperatures should and will be conducted to ascertain how effective elevated temperatures are in lessening the deleterious effects of irradiation.

Some data were presented which indicate that radiation sensitivity is dependent upon a number of factors such as grain size, structure and chemistry. Further study of such factors should be a fruitful field of investigation. The development of metals or alloys uniquely suited to use in intense radiation fields may be a result of such studies.

TABLE 1

Tensile Properties of Two Irradiated Alloy Steels

	Neutron Dose (nvt fast)	Yield Strength (psi)	Tensile Strength (psi)	Total Elongation* (%)	Uniform Elongation (%)
8½% Nickel Steel (Item 42)	0	91,800	119,000	29.5	21.5
	1.7×10^{19}	138,000	148,000	18	11
	1×10^{20}	183,000	184,000	5.5	2
High-Yield-Strength Alloy Steel (Item 60)	0	120,000	129,000	14	7.8
	1.7×10^{19}	170,000	171,000	4.5	0.5
	1×10^{20}	186,000	187,200	3.8	0.3

* Total elongation in 8 diameters; uniform elongation is elongation before onset of necking; strain rate $0.05\ min^{-1}$.

TABLE 2

Tensile Properties of Irradiated Coarse - and Fine-Grain Carbon Steel

	Neutron Dose (nvt fast)	Yield Strength (psi)	Tensile Strength (psi)	Total Elongation (%)	Uniform Elongation (%)
Coarse-Grain Steel (Item 55)	0	47,600	83,000	29	19
	1×10^{20}	116,000	121,000	6.5	1.5
Fine-Grain Steel (Item 56)	0	47,300	73,600	34	24.5
	1×10^{20}	96,500	102,000	11.5	4.5

* Total elongation in 8 diameters; uniform elongation is elongation before onset of necking; strain rate $0.05\ min^{-1}$.

TABLE 3

Tensile Properties of Irradiated High-Purity Iron (Item 25)

Subcritical Anneal

Strain Rate (min^{-1})	Neutron Dose (nvt fast)	Yield Strength (psi)	Tensile Strength (psi)	Total Elongation* (%)	Uniform Elongation* (%)
0.05	0	16,800	34,400	59.5	42
0.05	3.6×10^{19}	41,100	40,700	29.5	11
2.0	0	26,000	37,800	54	31
2.0	3.6×10^{19}	48,700	**	21	0

* Total elongation in 8 diameters; uniform elongation is elongation before onset of necking.

** Load decreased continuously after initial yielding.

TABLE 4

Tensile Tests on an Irradiated Carbon Steel Weldment (Item 65)

Material	Neutron Dose (nvt fast)	Yield Strength (psi)	Tensile Strength (psi)	Total Elongation[a] (%)	Uniform Elongation[a] (%)
SA-212 Base Plate	0	41,300	75,400	36	27
	1.7×10^{19}	91,600	98,000	11.6	5.8
	1×10^{20}	108,500	115,800	7	4
E-7016 Weld Metal	0	57,900	73,200	25.5	15.5
	0	59,000	71,700	24	15
	0	66,500	74,000	20.5	12.5
	5×10^{18}	69,300	77,500	18.5	10.5
	5×10^{18}	61,000	76,700	25	16.5[b]
	1.7×10^{19}	108,700	(c)	8	0
	1.7×10^{19}	106,500	(c)	8.5	0
	1×10^{20}	115,000	(c)	7.5	0

(a) Total elongation in 8 diameters; uniform elongation is elongation before onset of necking; strain rate, 0.05 min^{-1}.

(b) Irradiation temperature, this specimen only, was 600°F; all other specimens were irradiated at less than 200°F.

(c) Load decreased continuously after initial yielding.

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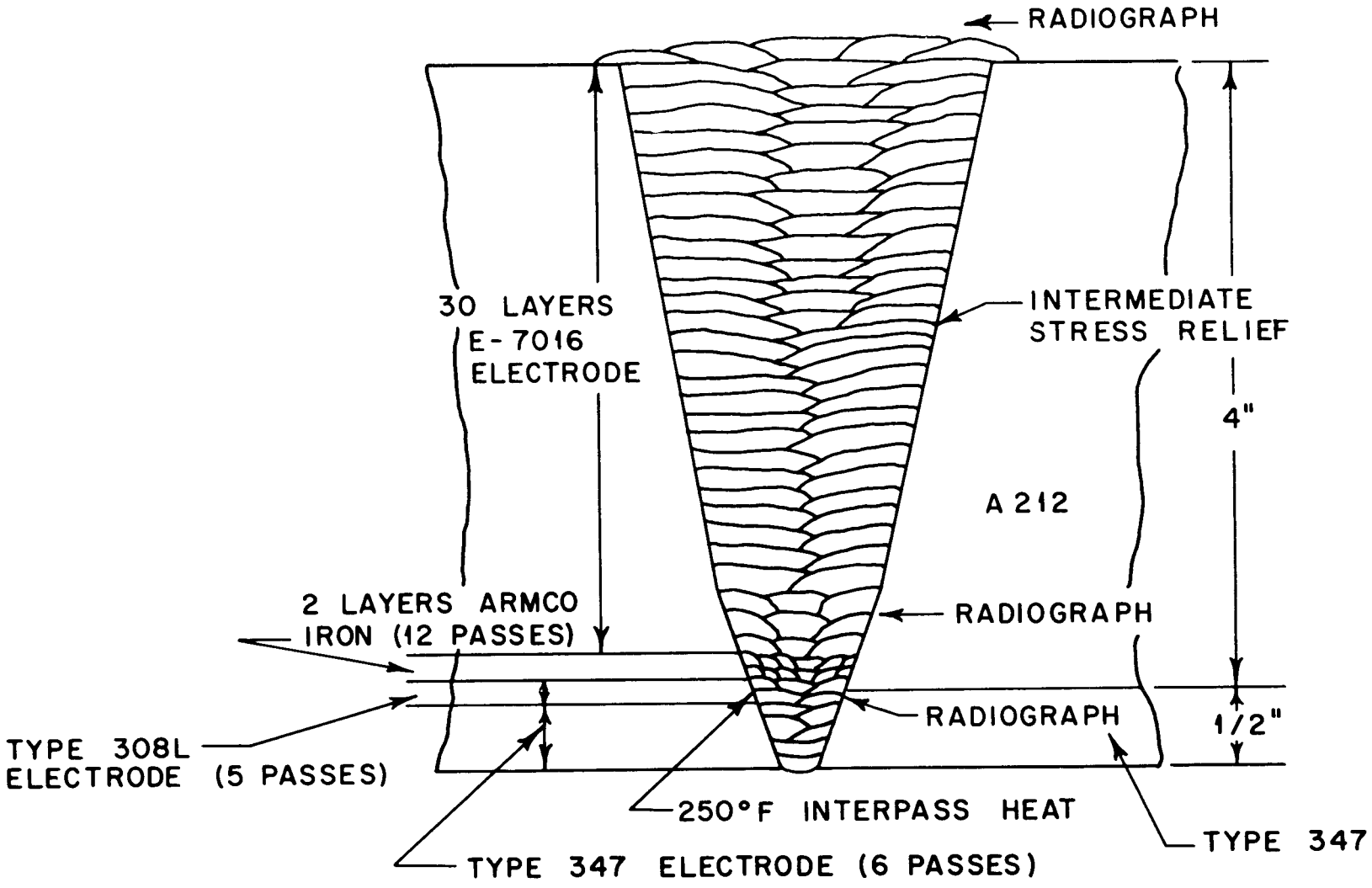

Fig. 1. Weld Design of Stainless Steel Clad Carbon-Silicon Steel Plate (Item 65).

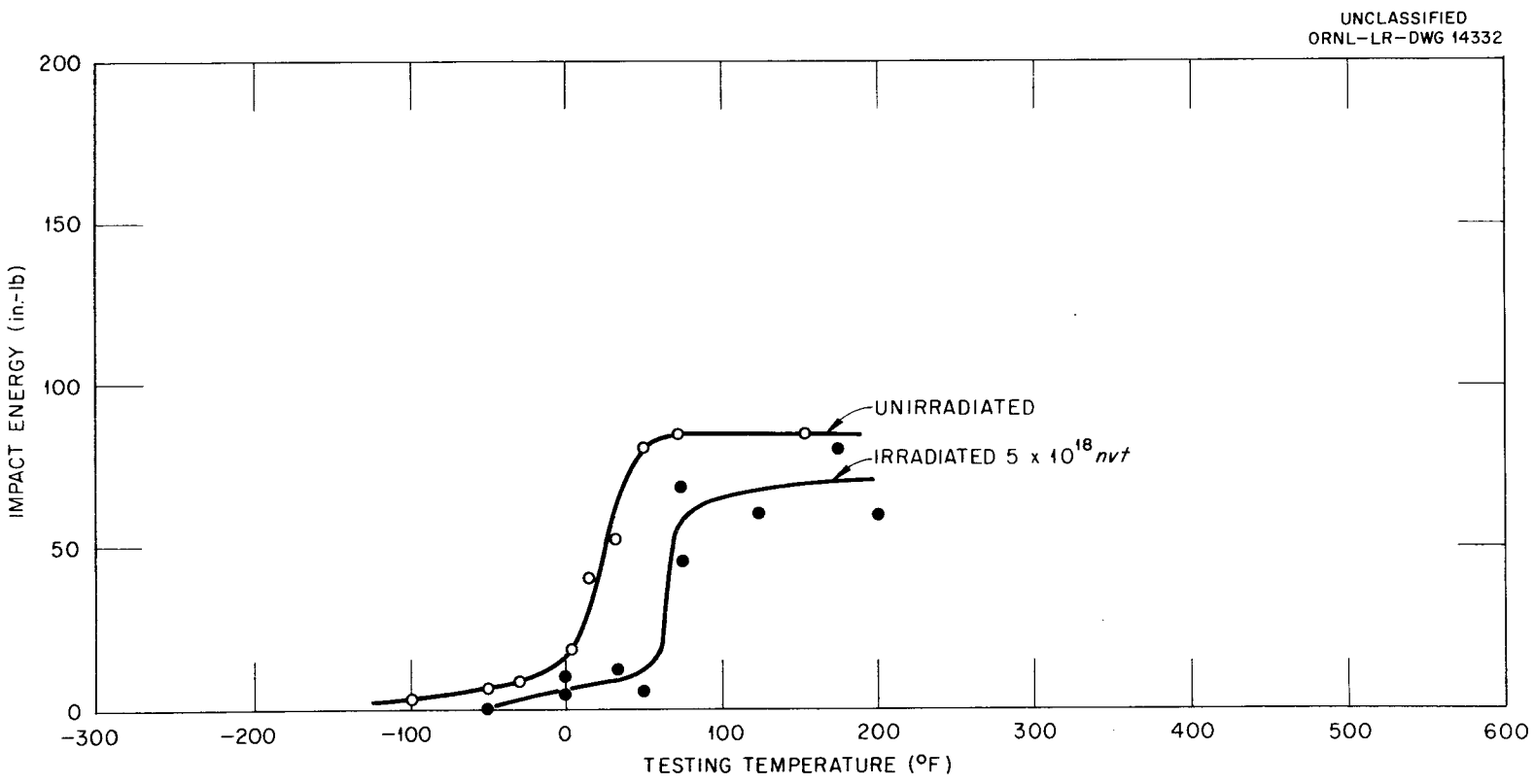

Fig. 2. Subsize Notch-Bar Impact Strength of Irradiated Hot-Rolled ASTM A-285-A Carbon Steel (Item 44).

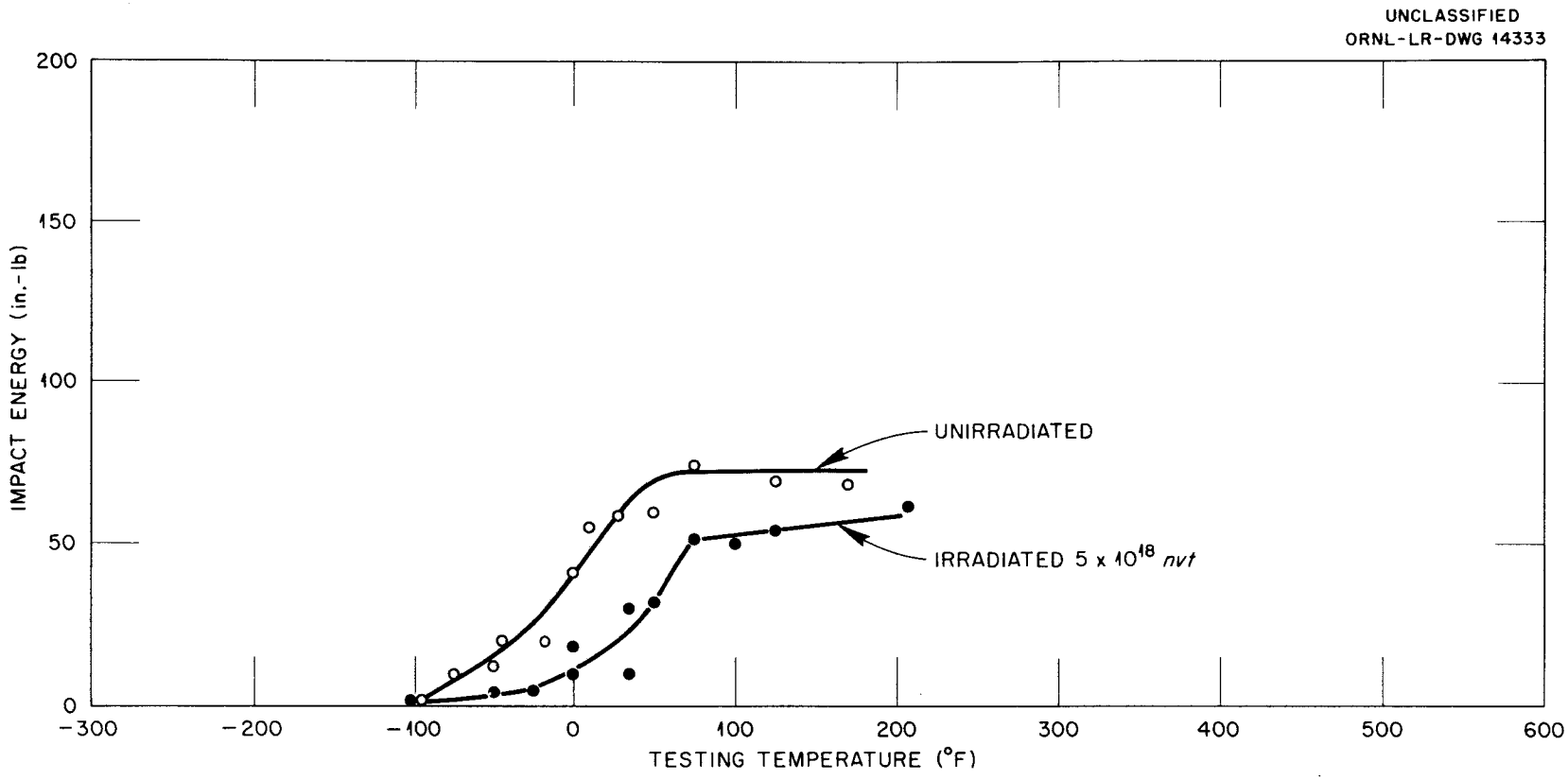

Fig. 3. Subsize Notch-Bar Impact Strength of Irradiated Normalized ASTM A-301-B Chromium-Molybdenum Steel (Item 45).

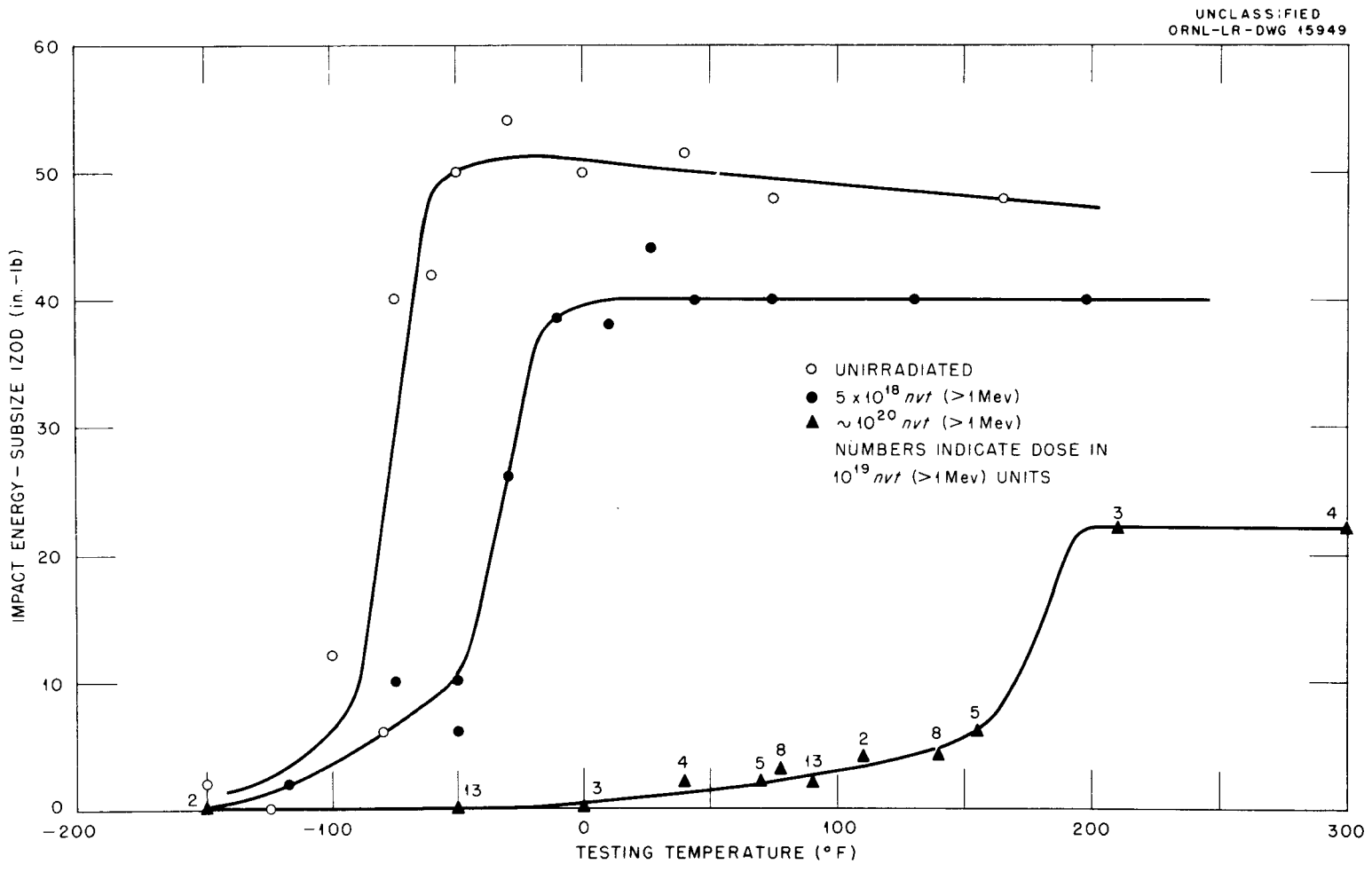

Fig. 4. Subsize Notch-Bar Impact Strength of Irradiated Hot-Rolled ASTM A-212-B Carbon-Silicon Steel (Item 43).

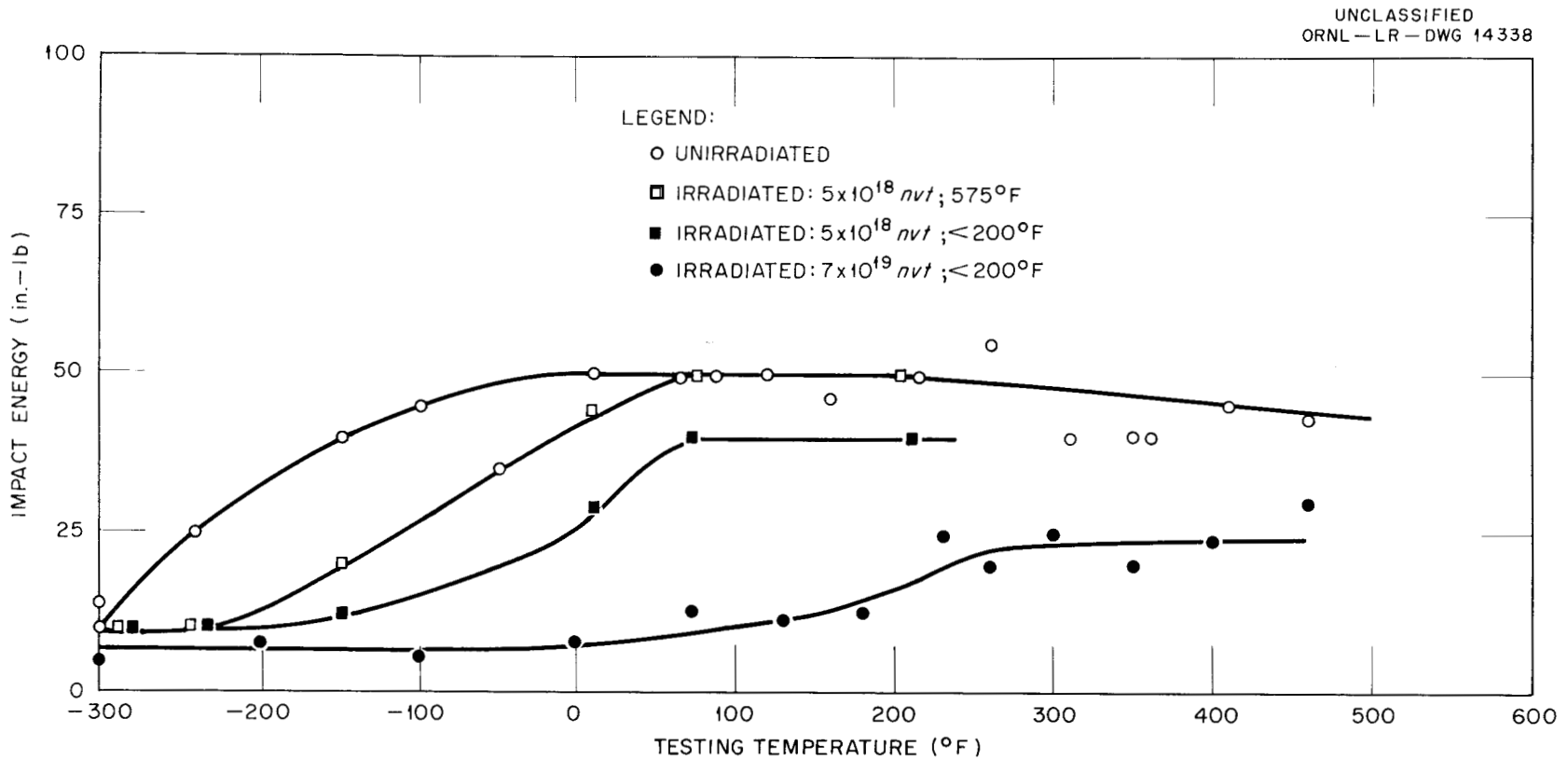

Fig. 5. Subsize Notch-Bar Impact Strength of an Irradiated High-Yield-Strength Alloy Steel, USS Carilloy T-1 (Item 60).

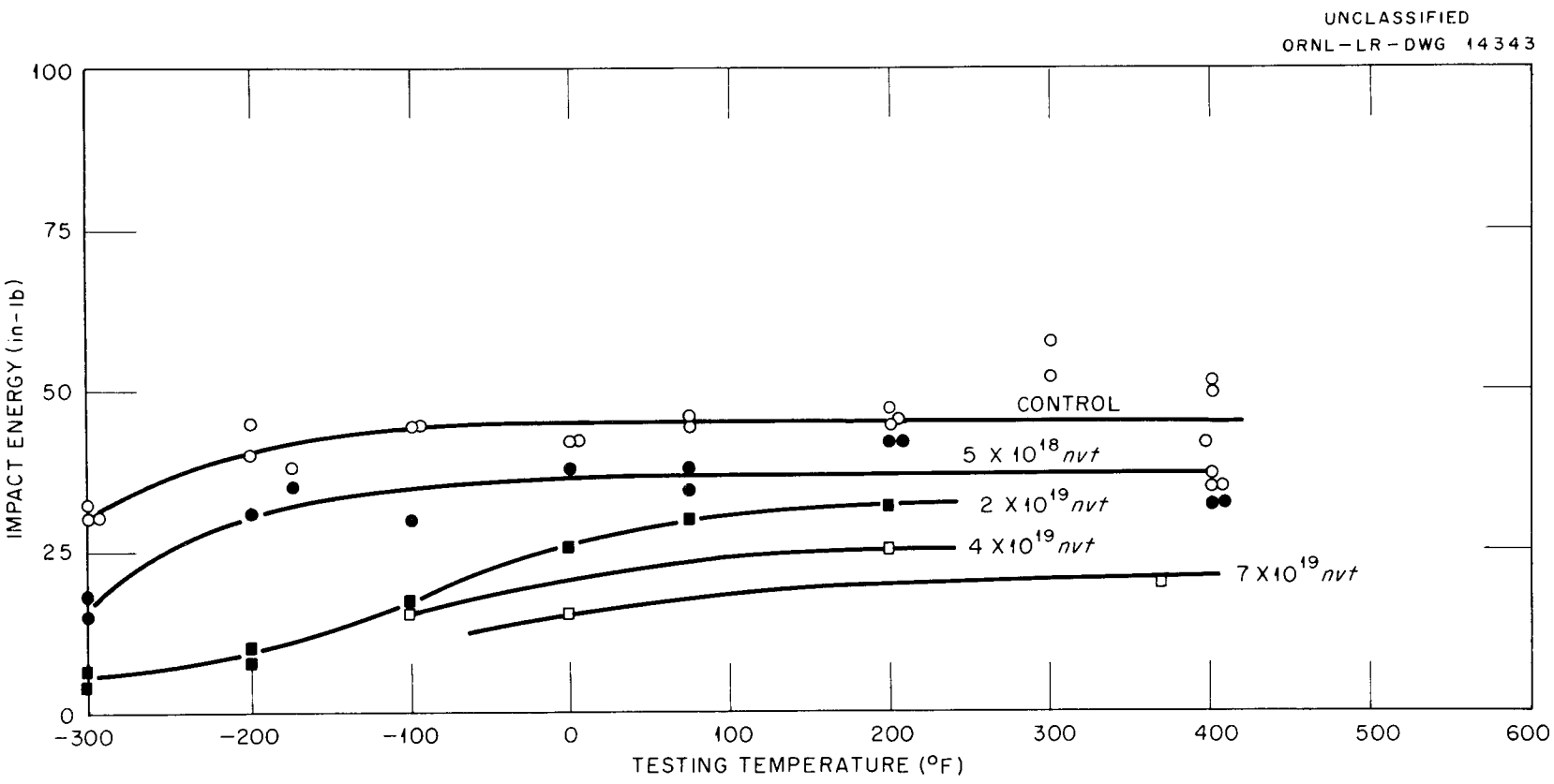

Fig. 6. Subsize Notch-Bar Impact Strength of Irradiated 8 1/2% Nickel Steel (Item 42).

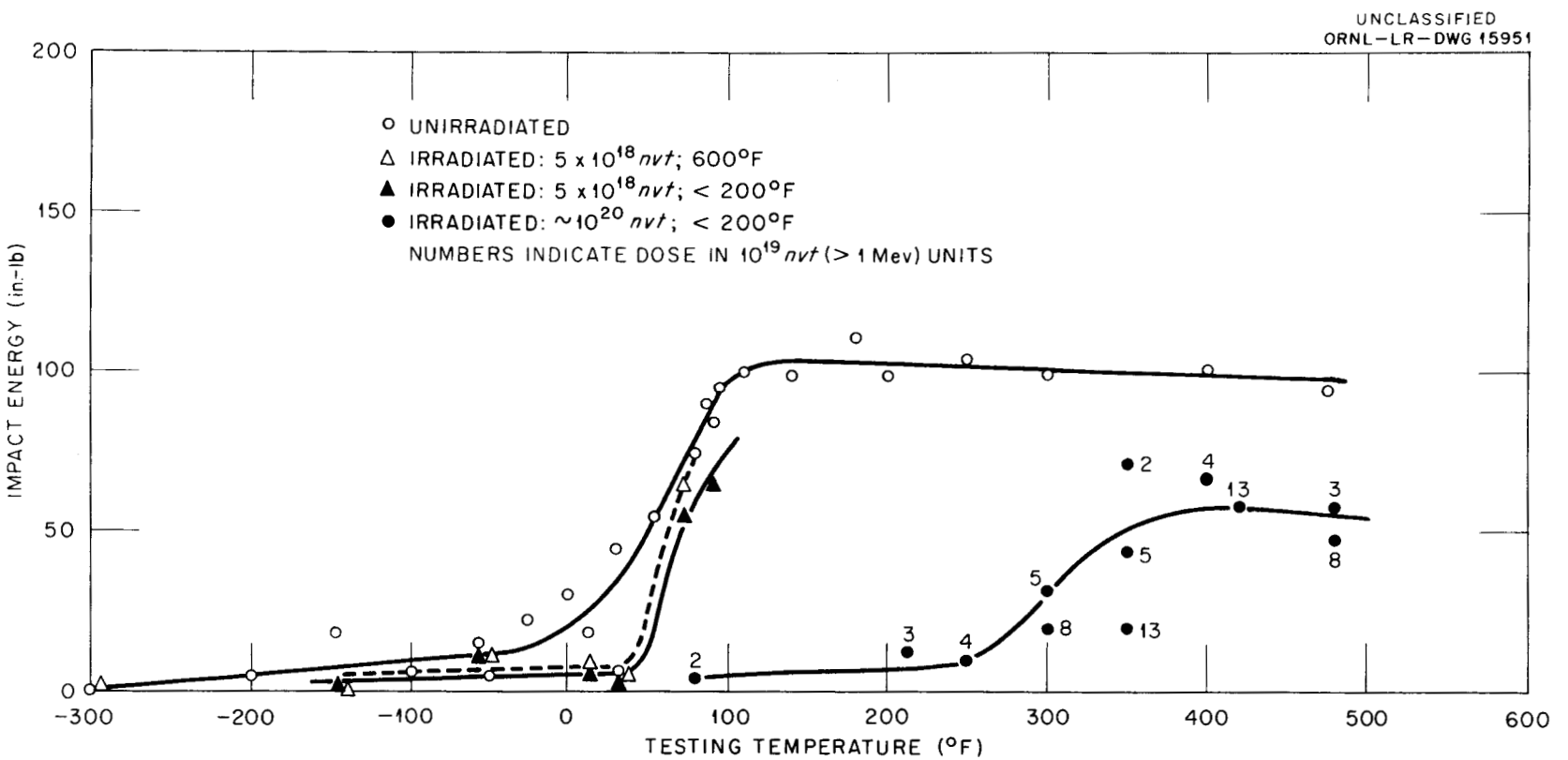

Fig. 7. Subsize Notch-Bar Impact Strength of Irradiated Coarse-Grain ASTM A-106-A Carbon Steel (Item 55).

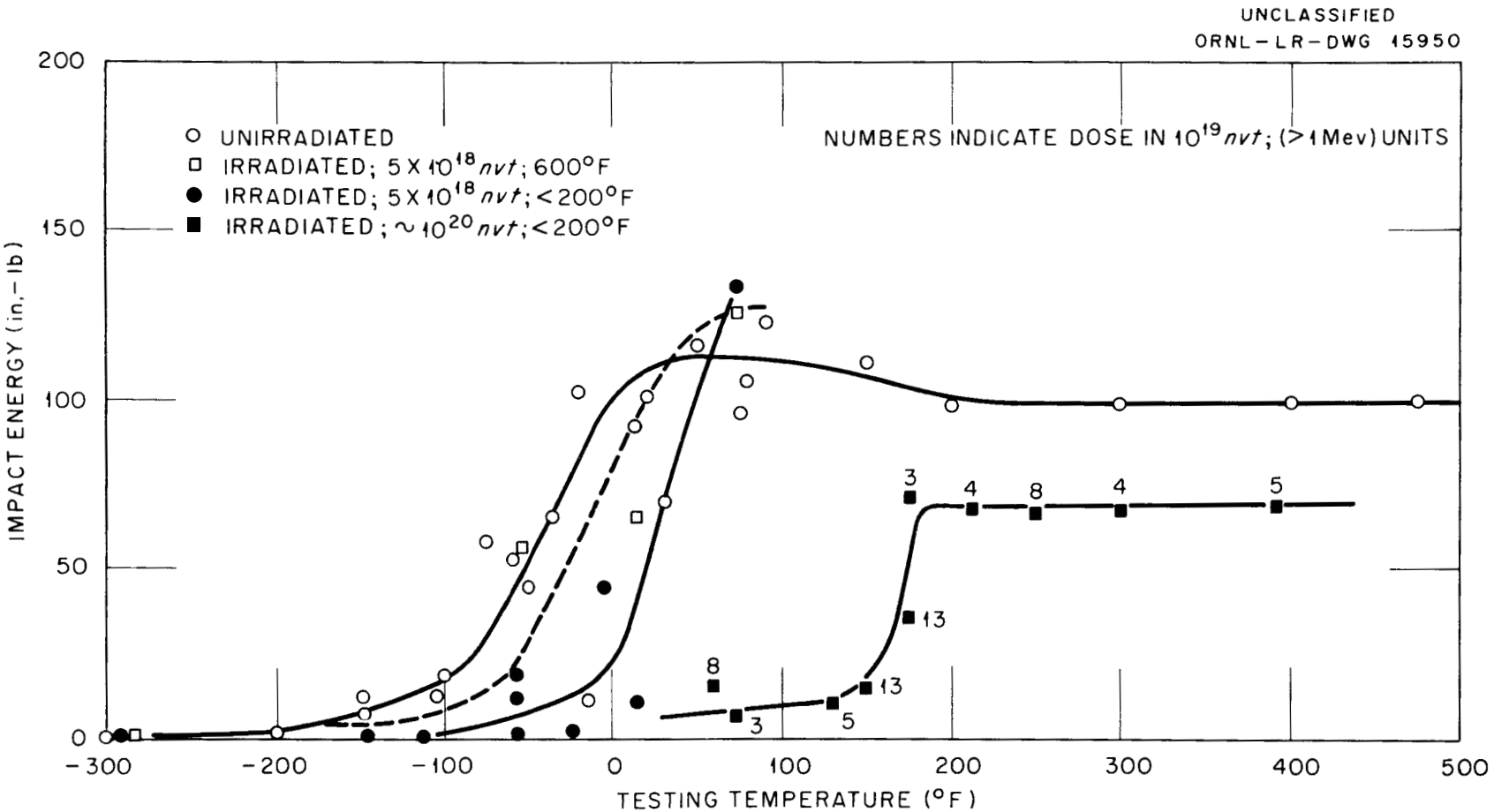

Fig. 8. Subsize Notch-Bar Impact Strength of Irradiated Fine-Grain ASTM A-106-A Carbon Steel (Item 56).

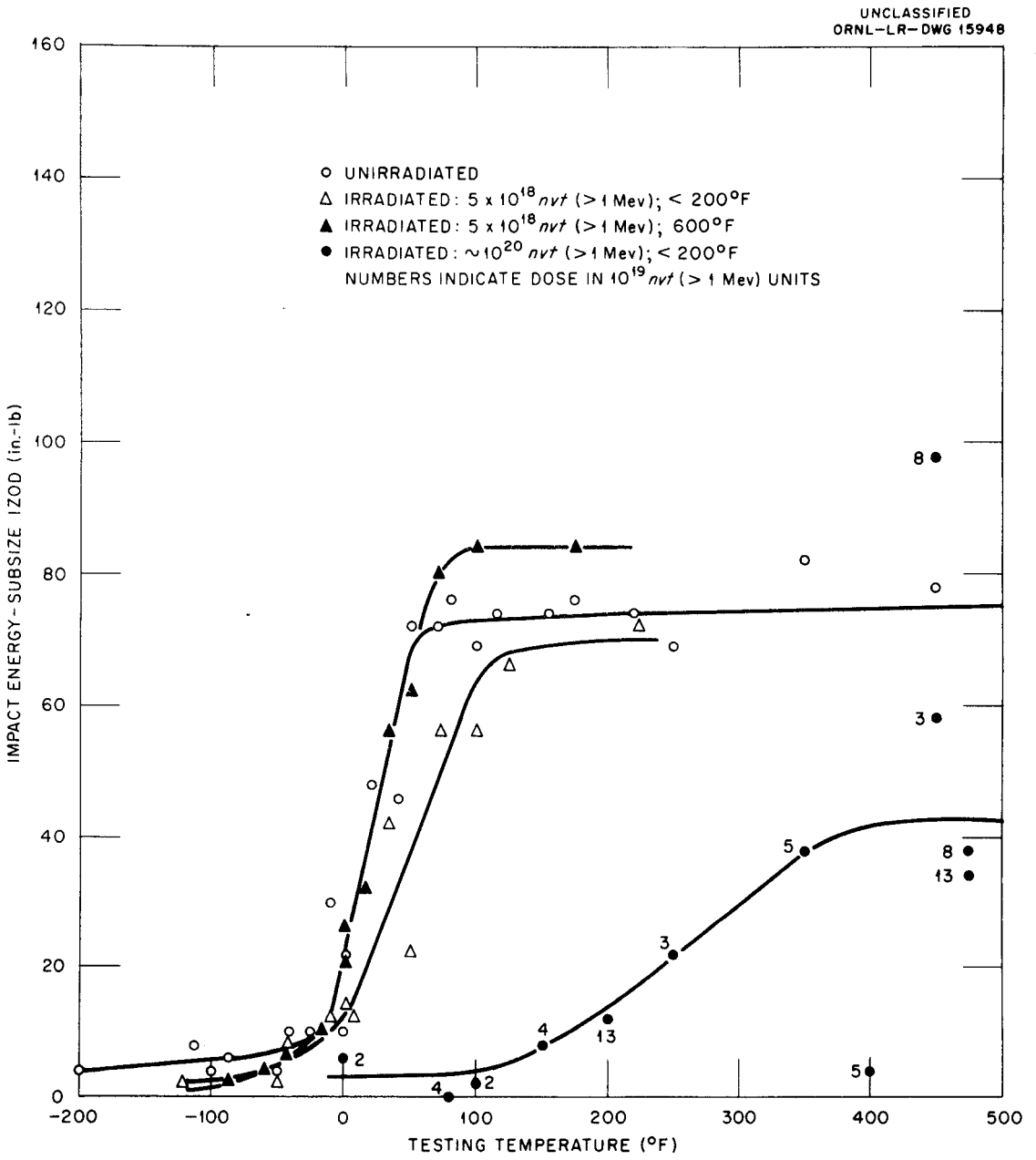

Fig. 9. Subsize Notch-Bar Impact Strength of Irradiated Fine-Grain ASTM A-212-B Carbon-Silicon Steel (Item 65, Base Plate).

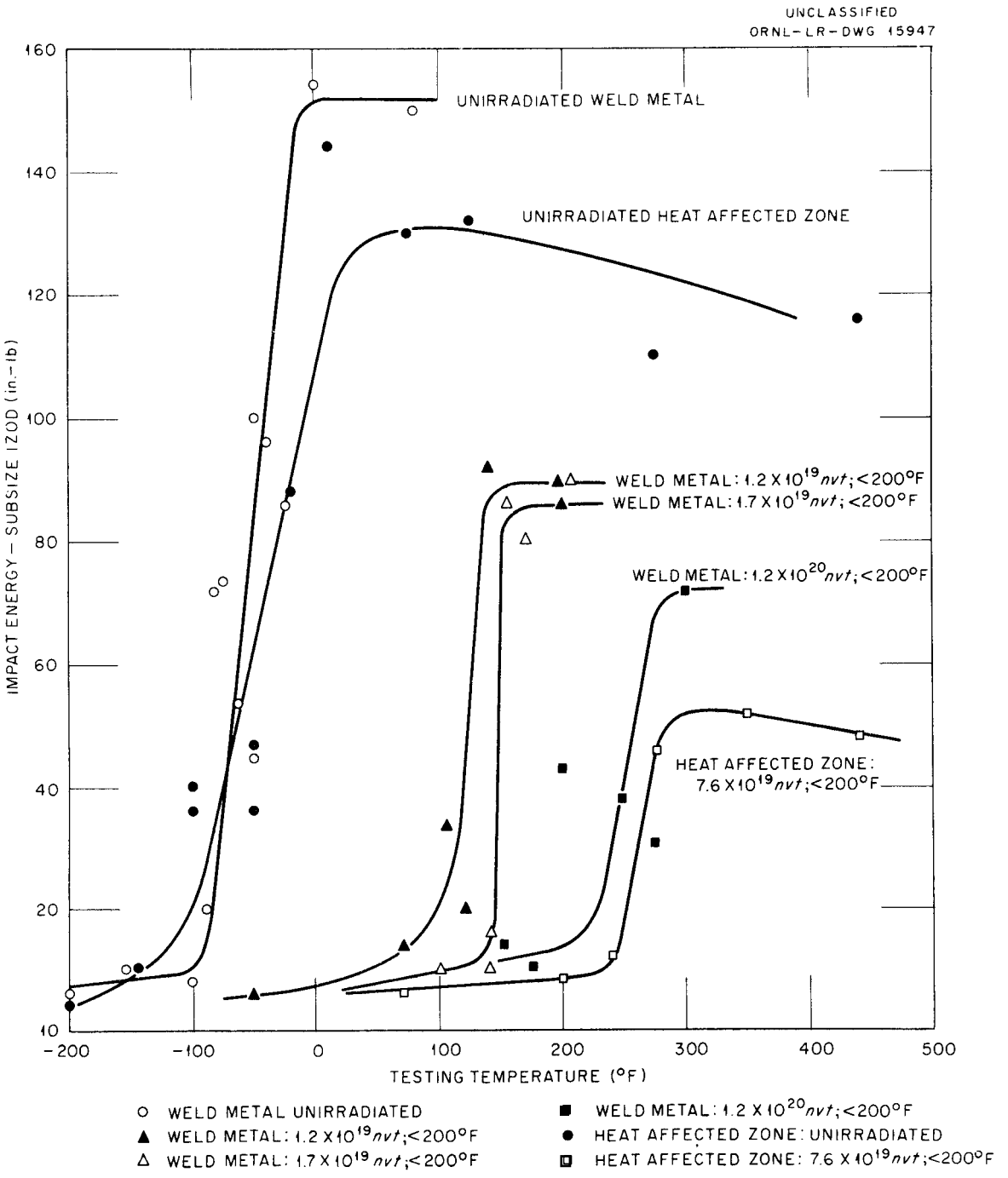

Fig. 10. Subsize Notch-Bar Impact Strength of Irradiated E-7016 Carbon-Steel Weld Metal and Heat-Affected-Zone of ASTM A-212-B Base Plate (Item 65, Weld and Heat-Affected-Zone).

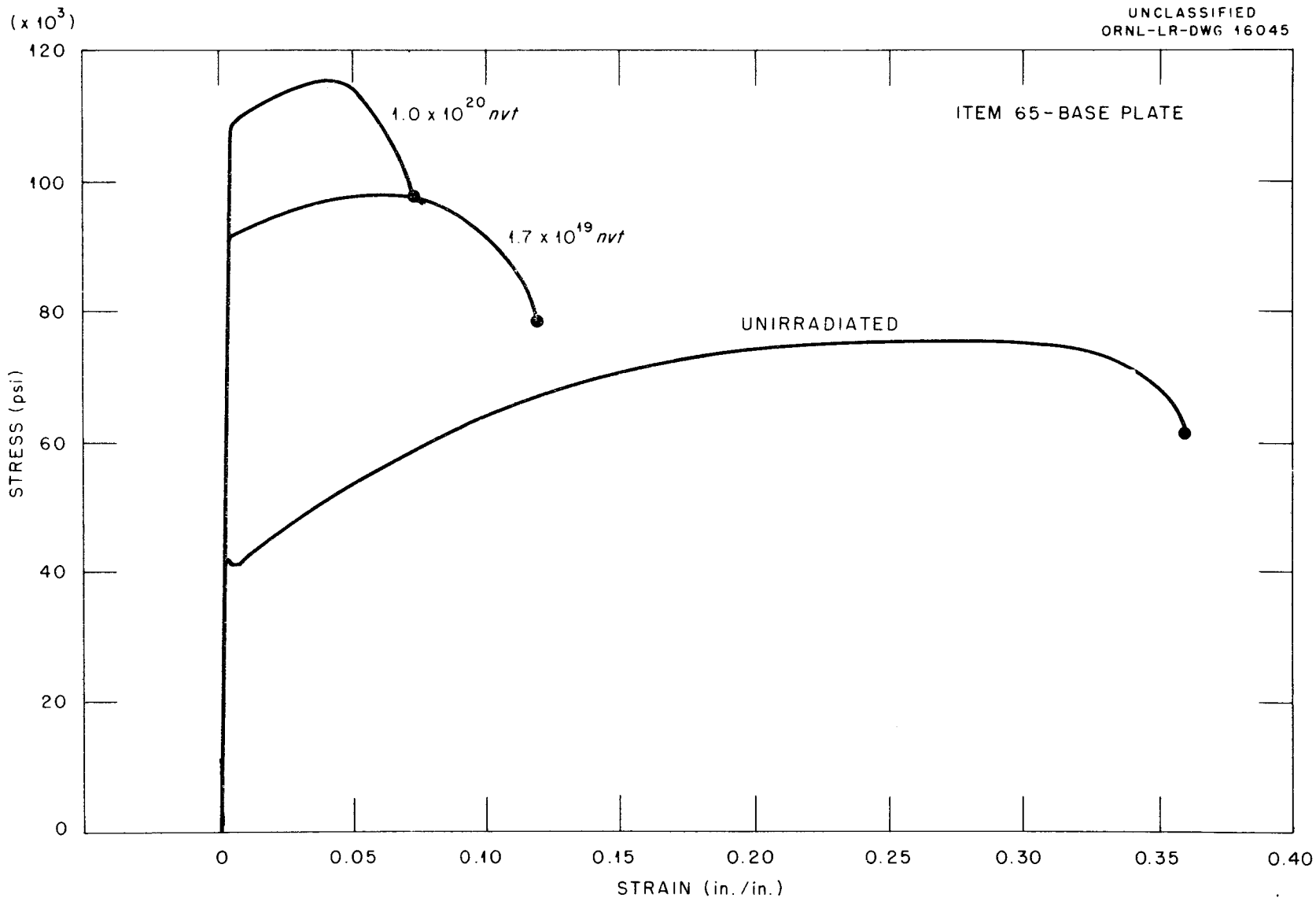

Fig. 11. Conventional Tension Stress-Strain Curves for Irradiated Fine-Grain ASTM A-212-B Carbon-Silicon Steel (Item 65, Base Plate).

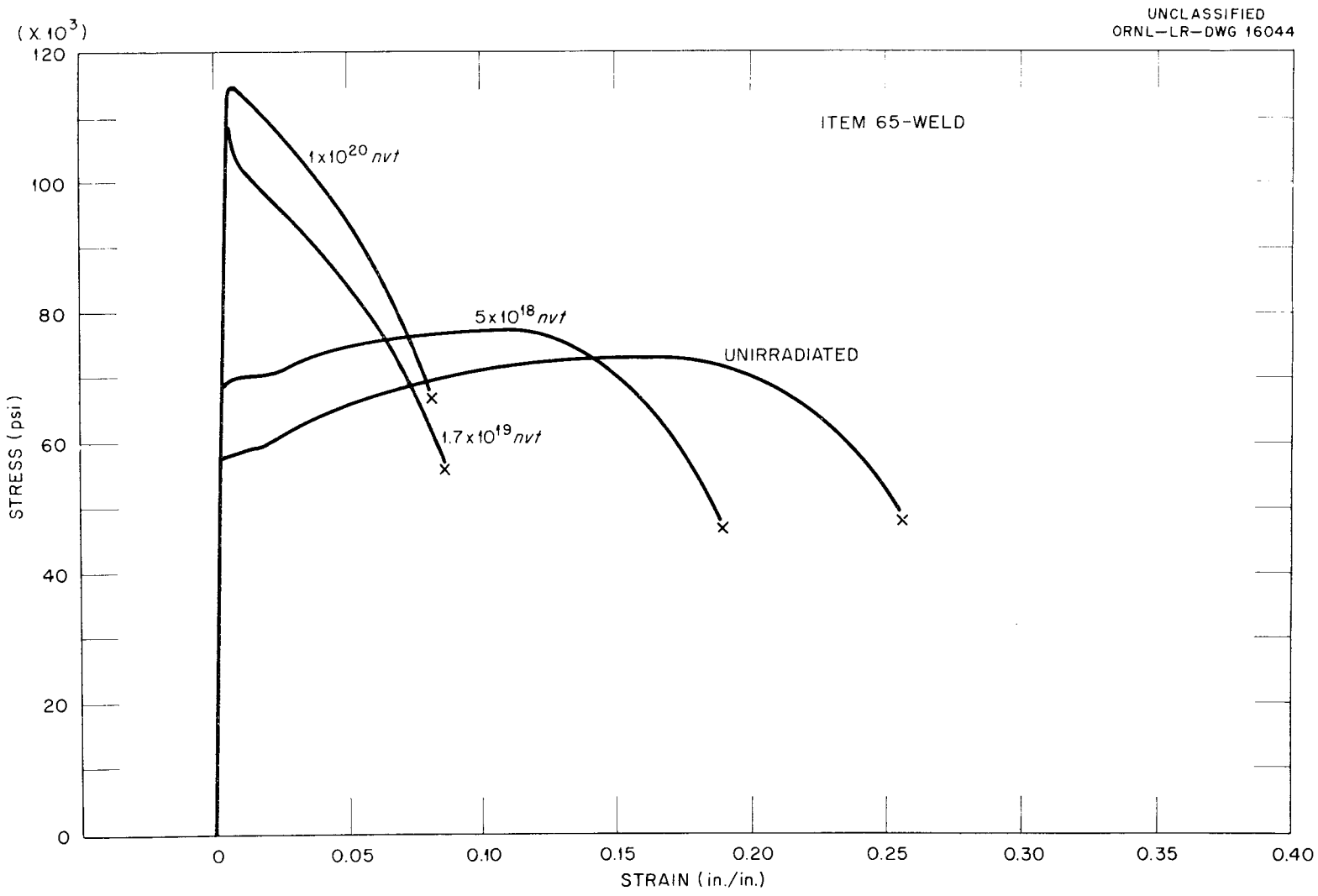

Fig. 12. Conventional Tension Stress-Strain Curves for Irradiated E-7016 Carbon-Steel Weld Metal (Item 65, Weld).