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STATUS OF KNOWLEDGE OF RADIATION EMBRITTLEMENT IN USA REACTOR P--ETC(U)

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Status of Knowledge of Radiation Embrittlement in USA Reactor Pressure Vessel Steels

STATUS OF KNOWLEDGE OF RADIATION EMBRITTLEMENT IN USA REACTOR PRESSURE VESSEL STEELS

Prepared by J. R. Hawthorne

Naval Research Laboratory

Prepared for
U.S. Nuclear Regulatory Commission

FEBRUARY 1982

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ABSTRACT

Advances by experimental research in the USA toward an improved understanding of property changes in steel by elevated temperature ($\sim 288^{\circ}C$) irradiation are summarized. Four areas of investigation are reviewed including the confirmation and demonstration of guidelines for radiation resistant steels, the isolation of metallurgical factors contributing to variable radiation embrittlement sensitivity, the qualification of in situ heat treatments for periodic vessel embrittlement relief, and the correlation of notch ductility and fracture toughness changes with irradiation.

Overall, the current state of the art provides both a high capability for tailoring steels for radiation service in new vessel construction and a promising method for controlling radiation embrittlement buildup in existing vessel construction.

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PREFACE

The objective of this document is to provide an overview of recent USA radiation effects investigations on reactor vessel steels and primary research accomplishments. The report was prepared at the invitation of the International Atomic Energy Agency and was one of a group of reports to the 1981 Specialists' Meeting on Irradiation Embrittlement and Surveillance of Reactor Pressure Vessels.

STATUS OF KNOWLEDGE OF RADIATION EMBRITTLEMENT IN USA REACTOR PRESSURE VESSEL STEELS

A. INTRODUCTION

Recent USA studies on radiation embrittlement development in pressure vessel materials have focused on four areas of investigation. One area of research effort was the confirmation and demonstration of the metallurgical requirements for improved (radiation resistant) steels. Primary questions addressed were the adequacy of new material specifications and guidelines and the ability of current technology to routinely produce highly radiation resistant plates, forgings and weld deposits. Efforts in the other areas of study were in support of early (pre-1972) pressure vessel construction and had the common objective of improving the understanding of radiation embrittlement behavior and its control. Here, one series of investigations probed variable radiation sensitivity factors in depth. A second series evaluated postirradiation heat treatment as a promising method of embrittlement relief. A third group of continuing studies investigated the correlation of notch ductility and fracture toughness changes with irradiation. In this case, the intent was to improve the understanding of the engineering significance of notch ductility changes with neutron exposure and to enhance the usefulness of currently available notch ductility data banks for Code and Standards applications.

The purpose of this report is to present an overview of the USA studies in the primary areas of investigation and to summarize the main observations and determinations.

B. DEMONSTRATION TESTS OF IMPROVED STEEL PRODUCTION

The radiation resistance of steels produced overseas was the general focus of attention in recent demonstration test studies. Interest was prompted by the use of these steels in certain USA reactor vessels and the concern that the use of raw materials from sources other than those employed in USA steel production could introduce different impurity element concentrations (or ratios) with a subsequent impact on radiation sensitivity characteristics.

Supplemental USA specifications for improving the irradiation serviceability of steels and welds place greater restrictions on allowable contents of copper, phosphorus, sulfur and vanadium impurities than the parent, i.e., primary, specifications. The express intent of the copper and phosphorus restrictions is to improve radiation resistance whereas the intent of the sulfur and vanadium limitations is to elevate the preservice upper shelf level for a greater toughness reserve against irradiation degradation. Supplemental specifications for A533-B and A508 Class 2 and Class 3 steels limit the maximum copper content to 0.10%Cu (heat analysis) and the maximum phosphorus content to 0.012%P for best radiation resistance [1 and 2]. The merit of the supplemental specifications has been firmly established experimentally for USA steel production [3]. Experimental tests of comparable "low" copper, "low" phosphorus steels from overseas production however have been insufficient in number to permit a broad assessment of the adequacy of the USA supplemental specifications for these materials.

In October 1977, the IAEA International Working Group on Reliability of Reactor Pressure Components (IWG-RRPC) [4] initiated a research program having the objectives of demonstrating that (a) careful specification of reactor steels can eliminate the problem of potential steel failure due to neutron irradiation effects, and that (b) knowledge has advanced to the point where steel manufacture and welding technology can routinely produce steel vessels of high radiation resistance. Materials obtained for the program included plates, forgings and welds from the Federal Republic of Germany (FRG), France and Japan (Table 1).

The Naval Research Laboratory (NRL) is participating in the IWG-RRPC study with a special interest in comparing overseas "improved" production against USA production. Its initial irradiation evaluations of the materials using Charpy-V (C_v) and fatigue precracked Charpy-V (PCC_v) test methods for notch ductility and dynamic fracture toughness (K_J) were completed this year [5]. NRL observations on material transition temperature elevations (C_v-41J index) are compared in Figure 1 to prior observations on embrittlement susceptibility for USA materials [3]. NRL findings on K_J change with irradiation generally support the C_v observations and are discussed later.

In Figure 1 the IWG-RRPC program materials (0.01 to 0.07%Cu) are found to perform as well as the low copper ($\leq$0.10%Cu) materials representing improved USA production. Thus, the results comprise a successful demonstration test of the adequacy of the USA supplemental specifications. Equally important, the combined data add confidence to the use of NRC Regulatory Guide 1.99 [6] for predicting radiation embrittlement to low copper content vessel material produced overseas.

An A533-B steel plate (HSST 03) representing USA melt practice was also included in the IWG-RRPC material investigations as a reference. Results for this plate are shown in Figure 1 and illustrate well the detrimental effect of a 0.12%Cu content compared to 0.01 to 0.07%Cu contents.

C. INVESTIGATIONS OF VARIABLE RADIATION SENSITIVITY

One USA study (NRL) investigated the interaction of nickel alloying and copper impurities in radiation sensitivity development [7]. An interaction first became suspect from relative embrittlement trends for high nickel, high copper content and low nickel, high copper content welds. Additional indications were found in summary data comparisons for A533-B steel (0.4 to 0.7%Ni) and A302-B steel ($\ll$0.4%Ni) [9]. Because high nickel content welds with low copper contents can show good radiation resistance as illustrated in Figure 2, a direct contribution of nickel (up to 1%Ni) to material radiation sensitivity has been discounted [3].

Table 1 - IWG-RRPC Program Materials

Material	Supplier	Source	Code	Composition (wt-%) Cu	P	Ni	S	V
S/A Weld	FRG	Thyssen-Maschinenbau	GW	0.03	0.011	0.93	0.009	0.01
A533-B Class 1	France	Marrel	FP	0.03	0.007	0.65	0.002	-[a]
A508 Class 3	France	FRAMATOME	FF	0.07	0.009	0.69	0.008	0.01
S/A Weld	France	FRAMATOME	FW	0.05[b] / 0.06[c]	0.015 / 0.011	0.56 / 0.73	0.011 / 0.008	0.01 / 0.01
A533-B Class 1	Japan	Nippon Steel	JP	0.01	0.007	0.66	0.007	-
A508 Class 3	Japan	Japan Steel	JF	0.04	0.007	0.76	0.005	-
S/A Weld	Japan	Mitsubishi	JW	0.04	0.008	0.89	0.003	-
A533-B	Japan	-[a,b]	LG	-	-	-	-	-
A533-B Class 1	USA	Lukens	HSST 03 (3MU)	0.12	0.011	0.56	0.018	-

[a] not determined

[b] 2.5 mm analysis

[c] 4.0 mm analysis

[d] base plate for weld JW

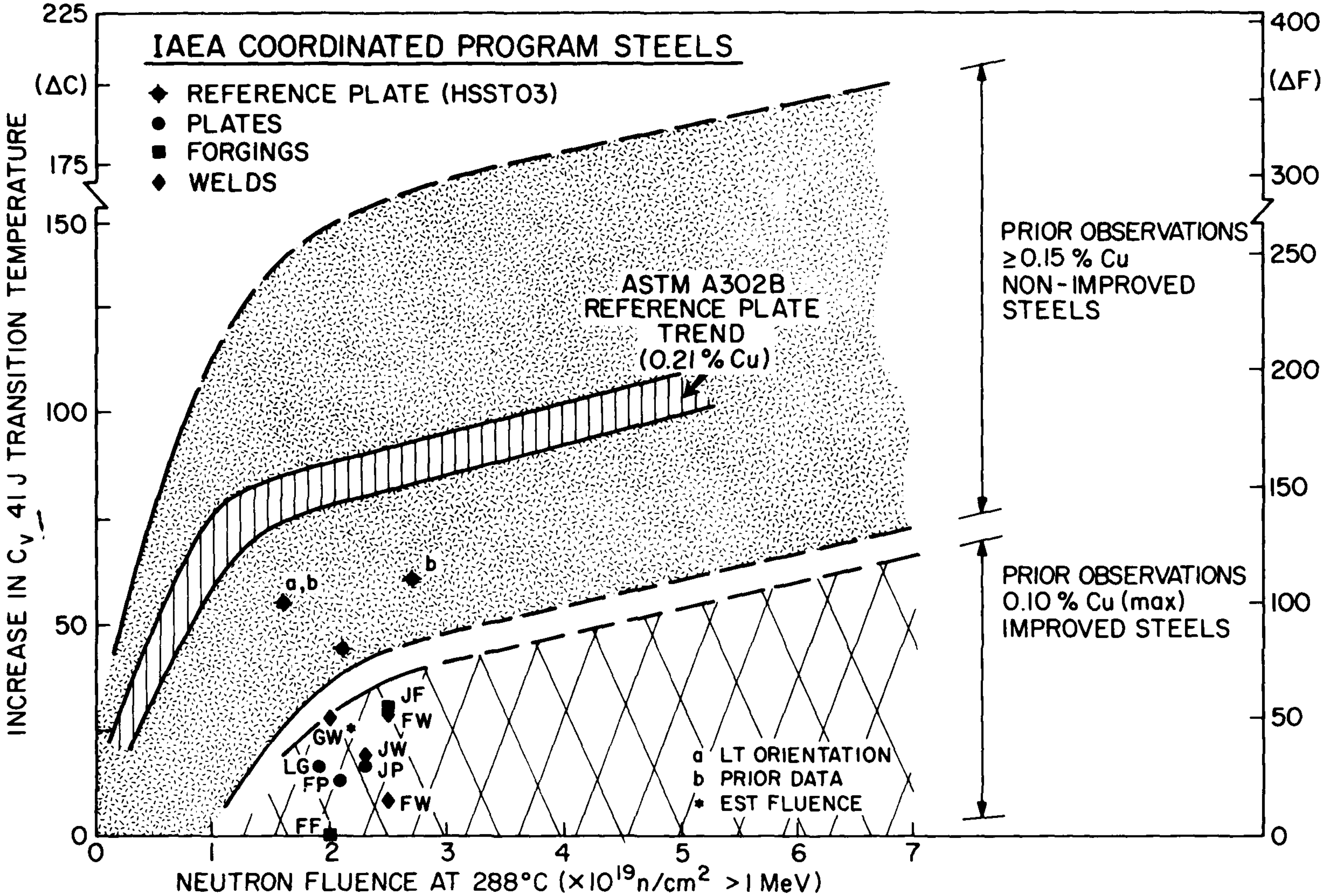

Fig. 1 - Comparison of radiation resistances of pressure vessel steels and welds produced by the FRG, France and Japan (0.01 to 0.07%Cu) with the trend behavior of improved steels (0.10%Cu max) produced in the USA. Good agreement is found. Data for a reference plate (HSST 03, 0.12%Cu) included in the IWG-RRPC study are also shown and illustrate the detrimental effect of a higher copper level on radiation resistance [5].

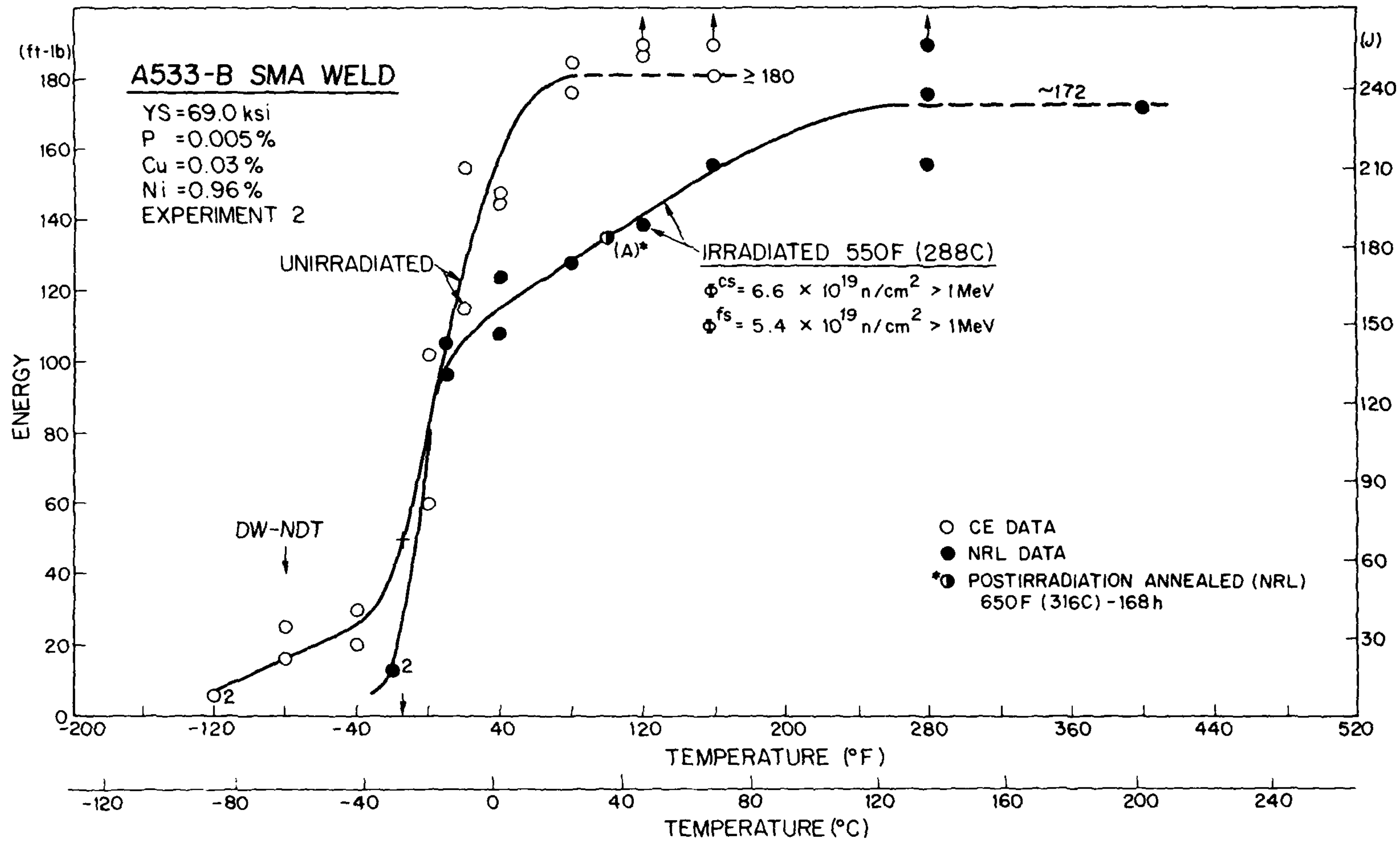

Fig. 2 - Notch ductility of a high nickel, low copper content shielded metal arc weld before and after 288°C irradiation to a high fluence [3].

Table 2 describes the materials matrix developed to test the interaction. The eight composition variations (A302-B base) were produced from two split laboratory melts. Plates rather than welds were studied for simplicity. Heat treatment cooling rates for the 12.7 mm thick plates simulated cooling rates of 152 mm thick plates at the quarter thickness for matching microstructures. C_v notch ductility properties of melt 6 are compared in Figures 3 and 4. Results of initial NRL comparisons of these material after 288C irradiation are given in Table 3 [7]. The neutron fluence (n/cm^2, E>1 MeV) was about 2.5×10^{19} n/cm^2. Referring to the data for plates 6B and 6C, a detrimental effect on radiation resistance of a 0.7%Ni content compared to a 0.28%Ni content is clearly shown. Likewise, the data for plates 5C and 5D from melt 5 indicate a greater transition temperature elevation with the greater of the two nickel contents (Table 4). The combined results are taken as a tentative confirmation of the suspect nickel-copper interaction. Irradiation assessments of the materials are continuing and include determinations of their relative response to postirradiation heat treatment (recovery). Additional (follow-on) investigations for the NRC are exploring the effects of combined copper, nickel and phosphorus contents as well as the effects of other impurity element-alloying element combinations on radiation sensitivity [10]. The material matrix for assessing the former is illustrated in Table 5; initial radiation comparisons are expected in early 1982.

Concurrent with the above, at least two reviews of data banks on irradiated steels and welds were made (or initiated) in the USA in the interest of identifying possible sensitivity factors by computer analysis. The Metal Properties Council (MPC) for example performed a survey and compilation of test reactor and power reactor (survillance) irradiation data for vessel steels that was available as of November 1977. Its report, "Prediction of the Shift in the Brittle/Ductile Transition Temperature of LWR Pressure Vessel Materials" now in publication clearly shows the importance of copper as a primary variable in radiation sensitivity development. Essentially a 1:1 relationship between C_v 68J and C_v 41J transition temperature increases by irradiatation was also found. The analysis concludes that the C_v 41J transition temperature elevation provides a more reliable means for measuring irradiation behavior than the C_v 68J transition elevation for the type steels surveyed. The ASTM Committee E10 on Nuclear Technology and Applications (Subcommittee E10.02) is in the process of developing a new (proposed) recommended practice for predicting neutron radiation damage to reactor vessel materials wherein one primary reference document will be the MPC survey report.

Independently, Combustion Engineering Corporation (CE) initiated an effort for the Electric Power Research Institute (EPRI) in late 1979, with objectives of maintaining and improving capabilities to predict the irradiation behavior of reactor vessel materials. The studies are building upon prior CE investigations by Varsik and Byrne [11] which evolved a model relating embrittlement susceptibility to material composition. The transition temperature relationship developed by their investigation is:

$$\Delta NDTT_{NORM} = F \text{ (Chemistry Ratio x Cu)}$$

where ΔNDT is the transition temperature normalized to a fluence of 3×10^{19} n/cm^2 and where the chemistry ratio is the value of:

Table 2 - Materials Matrix For Testing Ni-Cu Interaction
(Laboratory Split Melts; A302-B Base Composition)

		Composition (wt-%)[a]		
Melt	Cast Plate	Ni	Cu	Si
NRL 5	A	0.05[b]	0.05[b]	0.20
	B	0.30	0.05[b]	0.20
	C	0.30	0.15	0.20
	D	0.70	0.15	0.20
NRL 6	A	0.05[b]	0.30	0.20
	B	0.30	0.30	0.20
	C	0.70	0.30	0.20
	D	0.70	0.30	0.35

[a]Target value for melting operations

[b]Maximum value

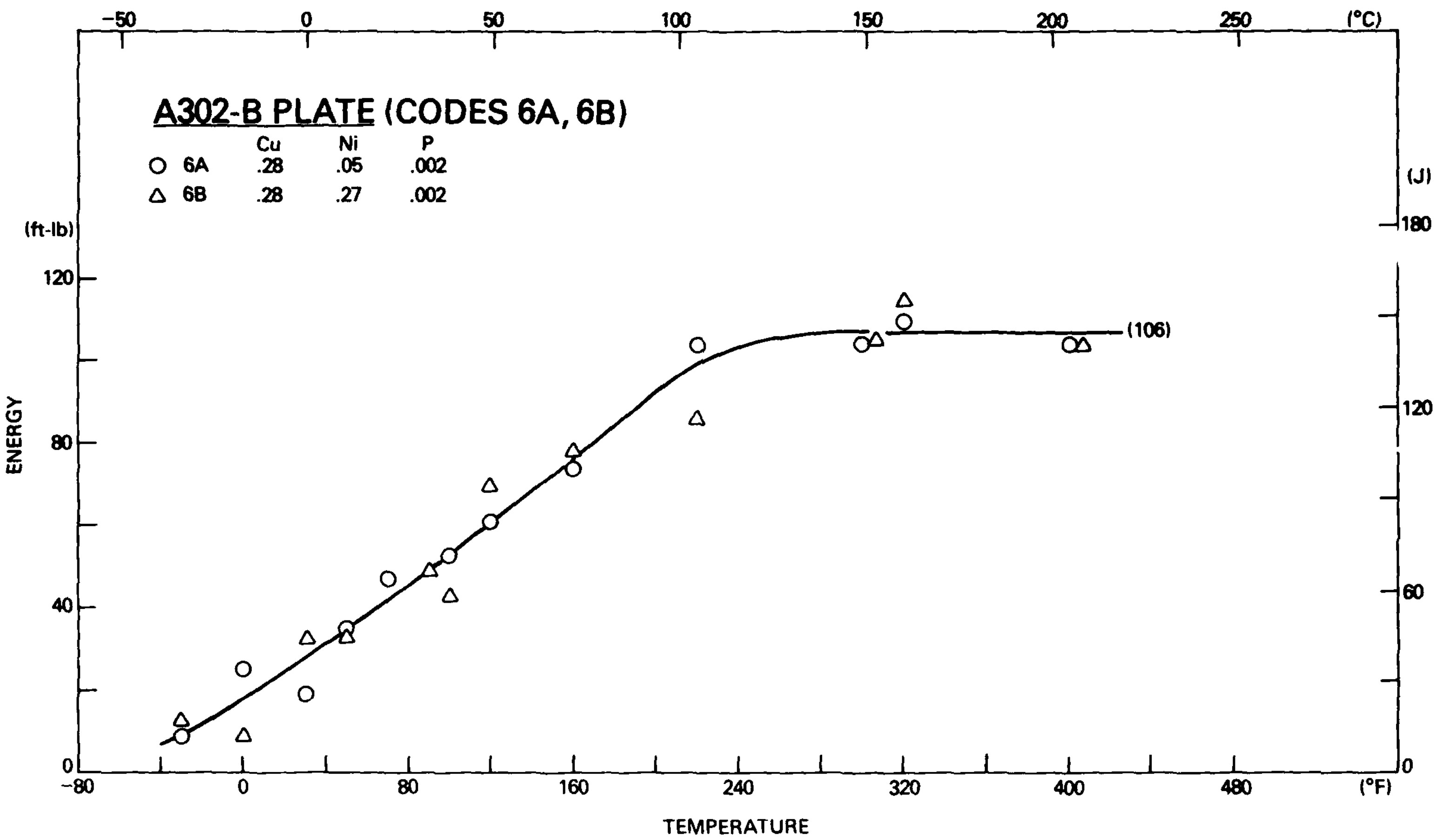

Fig. 3 - Charpy-V notch ductility of plates from the first and second casts from melt 6, demonstrating the agreement of properties.

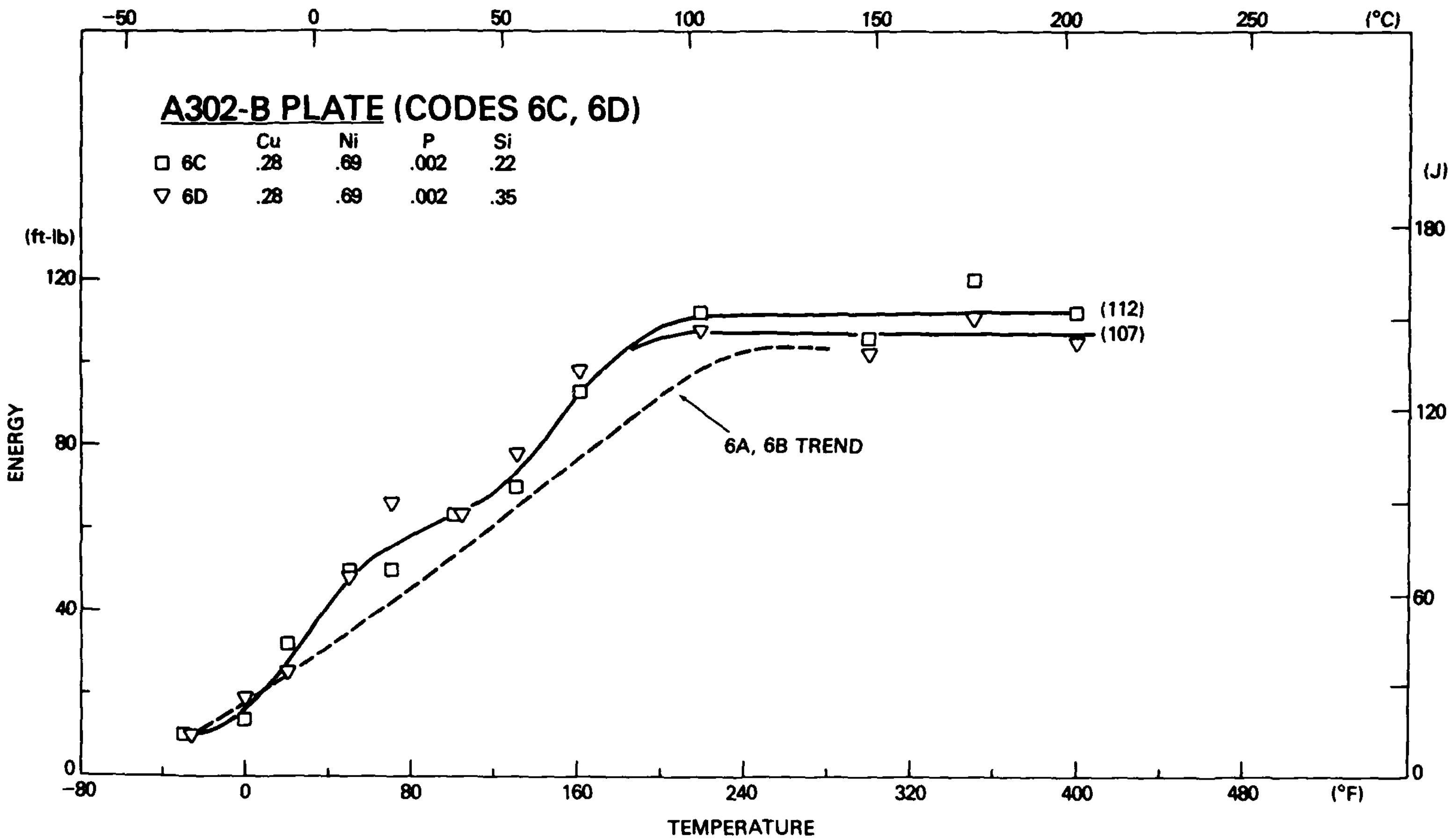

Fig. 4 - Charpy-V notch ductility of plates from the third and fourth casts from melt 6.

Table 3 - Postirradiation Notch Ductility Observations
(Melt NRL 6)

($\sim 2.6 \times 10^{19}$ n/cm^2 at 288°C)

Plate[a]	Ni (wt-%)	C_v 41J Increase (Δ $^\circ$C)	C_v Upper Shelf Decrease (Δ J)
6A	0.05	86	30
6B	0.30	81	30
6C	0.70	108	48
6C	0.70 (+0.35%Si)	103	41

[a]0.28% Cu, 0.22% Si

Table 4 - Postirradiation Notch Ductility Observations
(Melt NRL 5)

($\sim$2.4 x 10^{19} n/cm^2 at 288°C)

Plate[a]	Ni (wt-%)	C_v 41J Increase (Δ °C)	C_v Upper Shelf Decrease (Δ J)
5A	0.05	17	$\sim$ 0
5B	0.30	17	$\sim$ 0
5C	0.30 (+0.16% Cu)	64	$\sim$ 0
5D	0.70 (+0.16% Cu)	89	$\sim$ 0

[a] 0.005% Cu, 0.21% Si

Table 5 - Materials Matrix For Testing Ni-Cu-P Interaction[a]

(Laboratory Split Melts; A302-B Base Composition)

Melt Number	Cast/Plate Number	Composition (wt. - %)[b] Ni	Cu	P
7	A	0.70	0.05	0.005
	B	0.70	0.05	0.015
	C	0.70	0.05	0.026
8	A	0.70	0.30	0.005
	B	0.70	0.30	0.015
	C	0.70	0.30	0.026

[a]Cooperative effort by NRL and HEDL

[b]Target value for melting operations

$$\text{atomic percent}\left[\frac{1.5Ni + Si + 0.5C - 0.5\ (Mn - 0.5)}{0.5 + 0.5Mo}\right] \times Cu$$

Note the contrast with the general equation for transition temperature elevation given by the NRC Regulatory Guide 1.99:

$$\Delta RT_{NDT} = \left[40 + 1000\ (\%Cu - 0.08) + 5000\ (\%P - 0.008)\right]\left[\Phi/10^{19}\right]^{1/2}$$

In this case the maximum value of transition temperature elevation is limited depending on the fluence level.

Finally recent studies of variable radiation sensitivity directed some attention to the possibility for a saturation of radiation embrittlement at neutron exposure levels expected in service. This possibility was first tendered by Westinghouse [12] on the basis of certain power reactor surveillance data and represents a departure from test reactor data trends with fluence. EPRI has pursued this question further and reported to the 1981 ASTM E10 Minisymposium on Structural Materials Irradiation Study Programs that, tentatively, it has concluded that A533-B steel and weldments containing nickel alloying do not saturate at the fluence levels of interest. Its analysis suggests, however, that the rate of material embrittlement under irradiation may be a function of time at temperature. Experimental irradiations aimed at fully resolving this important question have been undertaken by the NRC.

D. POSTIRRADIATION HEAT TREATMENT FOR EMBRITTLEMENT RELIEF

Postirradiation heat treatment (annealing) as a method for the periodic embrittle ment relief of reactor vessels is receiving increasing interest in the USA. The method offers one possible solution to high embrittlement levels projected for high copper content welds in several older reactor vessels and is being studied extensively by NRL for the NRC [13 and 14] and by Westinghouse for the EPRI [15].

Earlier investigations indicated that temperatures of 399°C or higher will be required if an anneal is to be sufficiently effective in terms of notch ductility recovery [16]. More recent efforts focused on material behavior upon return to service, i.e., after the anneal. Obviously, the ultimate test of the potential of the method rests with properties behavior under irradiation (I), annealing (A) and reirradiation (R) conditions.

NRL has reported the IAR performance of two weld deposits produced commercially and containing 0.35%Cu and 0.71%Ni [13,14]. The study was designed to test the ability of periodic 399°C-168 hour heat treatments to hold notch ductility changes below Code-allowable limits and to determine and compare material reembrittlement rates upon reirradiation. Two series of experiments have been conducted. The more recent series included compact tension (CT) specimens for fracture toughness (K_J) determinations by the single specimen compliance technique and J integral assessment procedures as well as C_v specimens.

In addition, selected specimen groups were carried through two full cycles of annealing and reirradiation. The results are summarized in Figures 5 and 6. The data trends with annealing and reirradiation versus the trends without annealing verify that the method can be very effective in reducing the build up of irradiation effects. "Embrittlement arrest" was also found in the IAR performance of the CT specimens [14].

Closer inspection of the C_v data reveals that the response of upper shelf properties to annealing, i.e., percent recovery, is different from that of transition temperature properties. Also, comparisons of notch ductility and tensile property trends with annealing and reirradiation reveal parallels between transition temperature change and yield strength change and between upper shelf change and tensile ductility change (see Table 6). Furthermore, the IAR data in Figures 5 and 6 show that the rate of embrittlement after annealing initially is greater than the rate of embrittlement of nonannealed material. The trends suggest that the "damage" most readily introduced into the material (that produced early in radiation service) is also that "damage" most readily removed by the anneal. This projection is based, in part, on the similarity of radiation embrittlement rates observed for the annealed material and the virgin material. In-depth studies of reembrittlement path are now underway.

E. CORRELATION OF FRACTURE TOUGHNESS CHANGE WITH IRRADIATION

Tentative correlations of notch ductility and fracture toughness change with 288°C irradiation are beginning to evolve from USA studies of relative C_v versus PCC_v and relative C_v versus CT test behavior [14 and 17].

Figure 7 presents a comparison of postirradiation transition temperature elevations indexed by the 41J temperature [C_v method] and the K_J 100 MPa$\sqrt{m}$ temperature (PCC_v method). The materials represented are the IWG-RRPC program steels [5] and the NRL-EPRI RP886-2 program steels [18]. Considering the number of specimens available for each test condition (limited), the independent measures of transition temperature change are in exceptionally good agreement (typically within 15°C). A slight bias toward a higher K_J 100 MPa$\sqrt{m}$ transition elevation by irradiation is seen overall. The primary exception to this general pattern of correspondence is a forging (EPRI Code BCB). In this case, the K_J data for preirradiation and postirradiation conditions (see Figure 8) depict wide scatter, making an estimation of average behavior difficult. Figure 9 provides the C_v data for the material for reference. Additional comparisons of 41J and 100 MPa$\sqrt{m}$ transition temperature elevations are expected from continuing programs.

One focus of evaluations with CT specimens is on fracture initiation toughness. However, pressure vessel materials normally will exhibit elastic-plastic behavior over a major portion of the brittle-to-ductile transition region. Accordingly, the evaluations are also characterizing the slow-stable crack extension phenomenon commonly described by the R curve (see Figure 10). The latter is useful not only for defining crack initiation but also for assessing the potential for crack instability [14]. The inferred toughness, K_{Jc}, is computed from the relation:

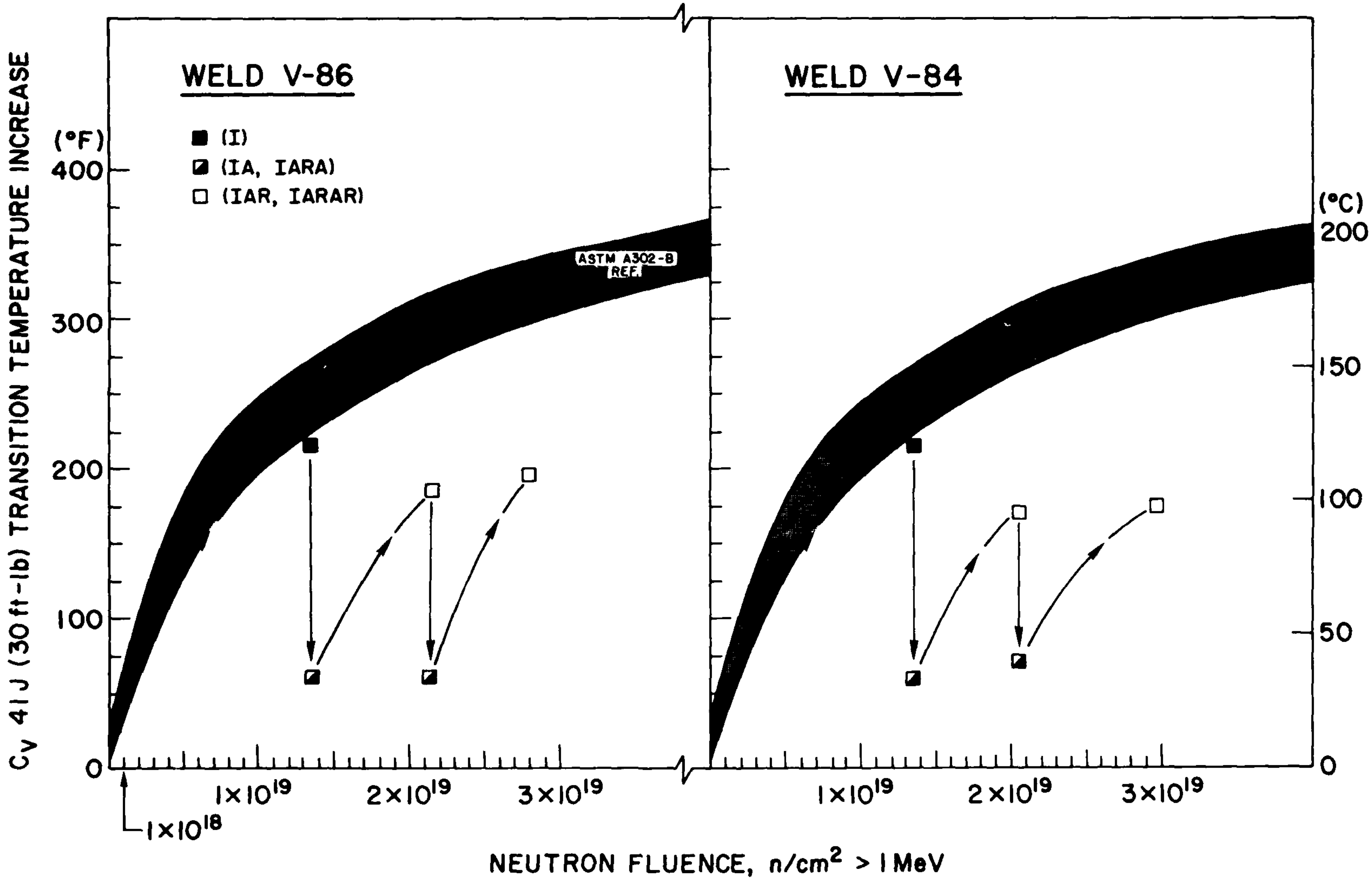

Fig. 5 - Transition temperature behavior of two submerged arc welds (0.35%Cu, 0.7%Ni) with 288°C irradiation followed by two cycles of 399°C annealing and 288°C reirradiation. The shaded band refers to a data trend for the ASTM A302-B reference plate (0.21%Cu, 0.18%Ni) with < 232°C irradiation; the lower boundary of the band appears to describe the 288C embrittlement trend of the welds without intermediate annealing [13].

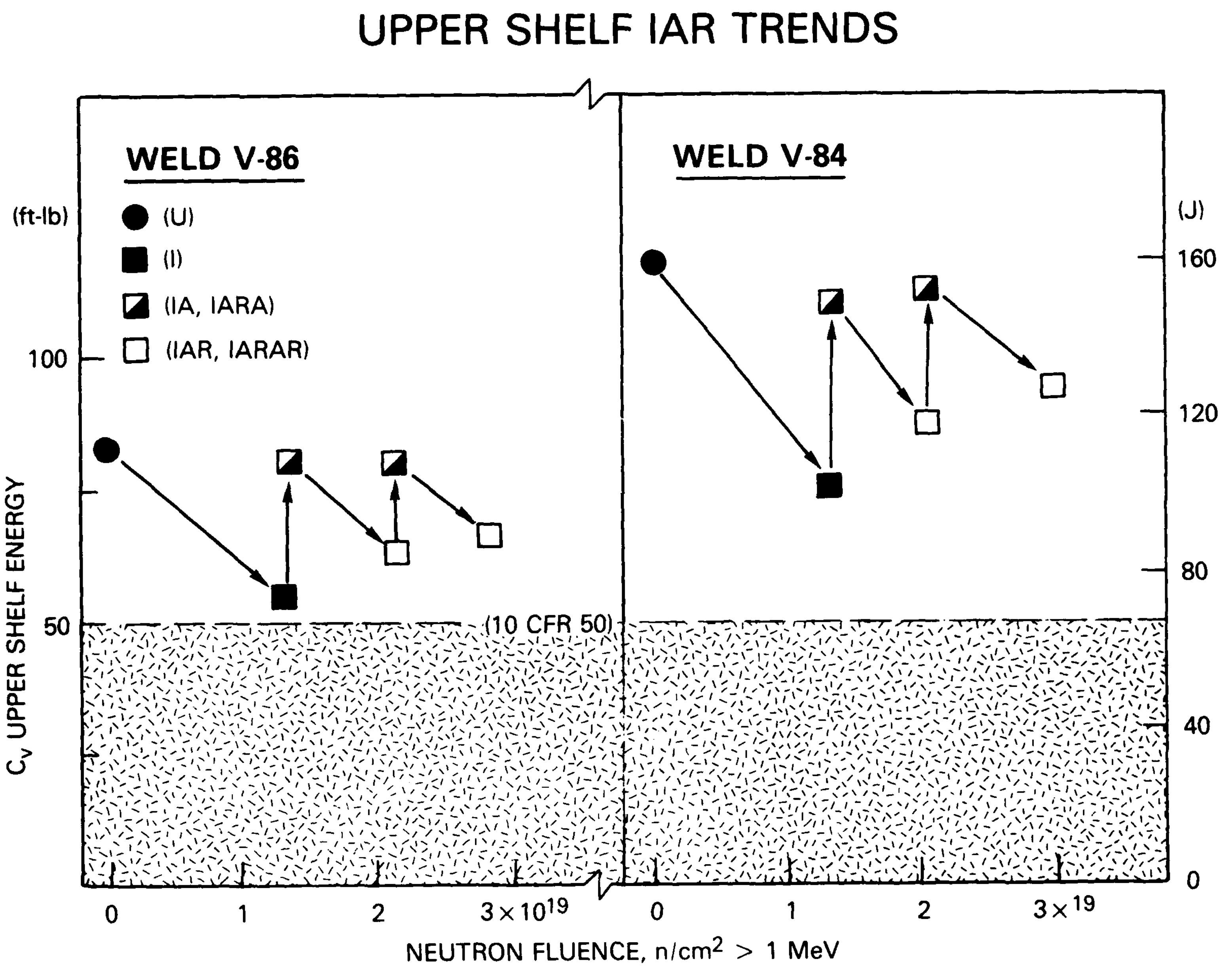

Fig. 6 - Charpy-V upper shelf behavior of two submerged arc welds (0.35%Cu, 0.7%Ni) with 288°C irradiation followed by two cycles of 399°C annealing and 288°C reirradiation. The shaded region indicates upper shelf levels that are not in conformance with the Code of Federal Regulations (10CFR50) [13].

Table 6 - Tensile Properties of Submerged Arc Welds (0.35%Cu, 0.7%Ni) with IAR [13]

Weld	Condition	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
V86	Unirradiated	74	94	23.9
	Irradiated	101	115	19.7
	IAR (2 cycles)	95	111	21.8
	Annealed 399°C (2 cycles)	91	107	24.2
V84	Unirradiated	74	92	23.5
	Irradiated	106	120	22.1
	IAR (2 cycles)	95	112	20.1
	Annealed 399°C (2 cycles)	84	102	23.6

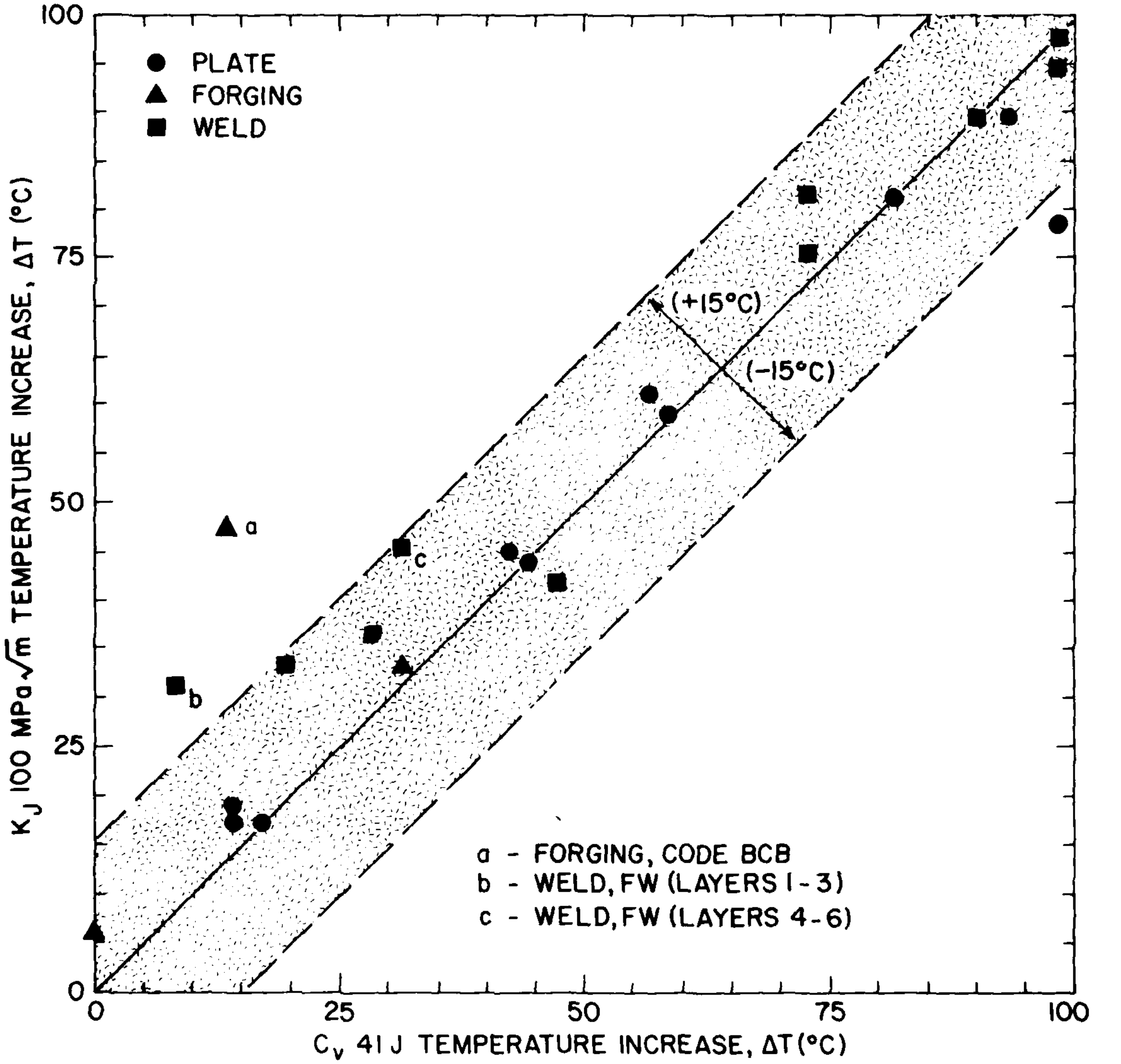

Fig. 7 - Comparison of K_J 100 MPa$\sqrt{m}$ and 41J transition temperature elevations by 288°C irradiation. Agreement within 15°C is observed for all but two data sets (Forging BCB and Weld FW, layers 1-3) [5,18].

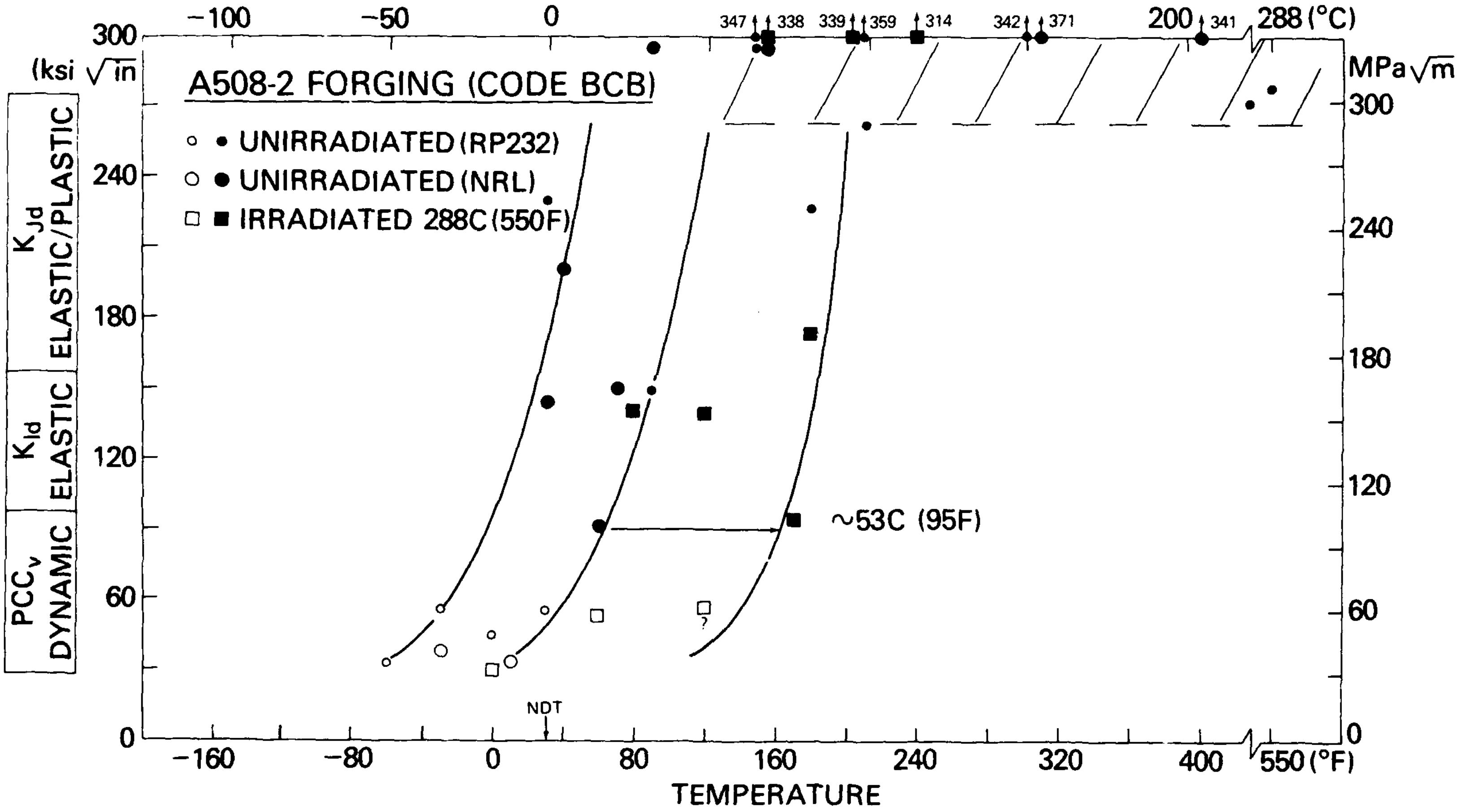

Fig. 8 - Dynamic fracture toughness of Forging BCB before and after 288°C irradiation (PCC_v test method). Note the wide scatter in data for each test condition [18].

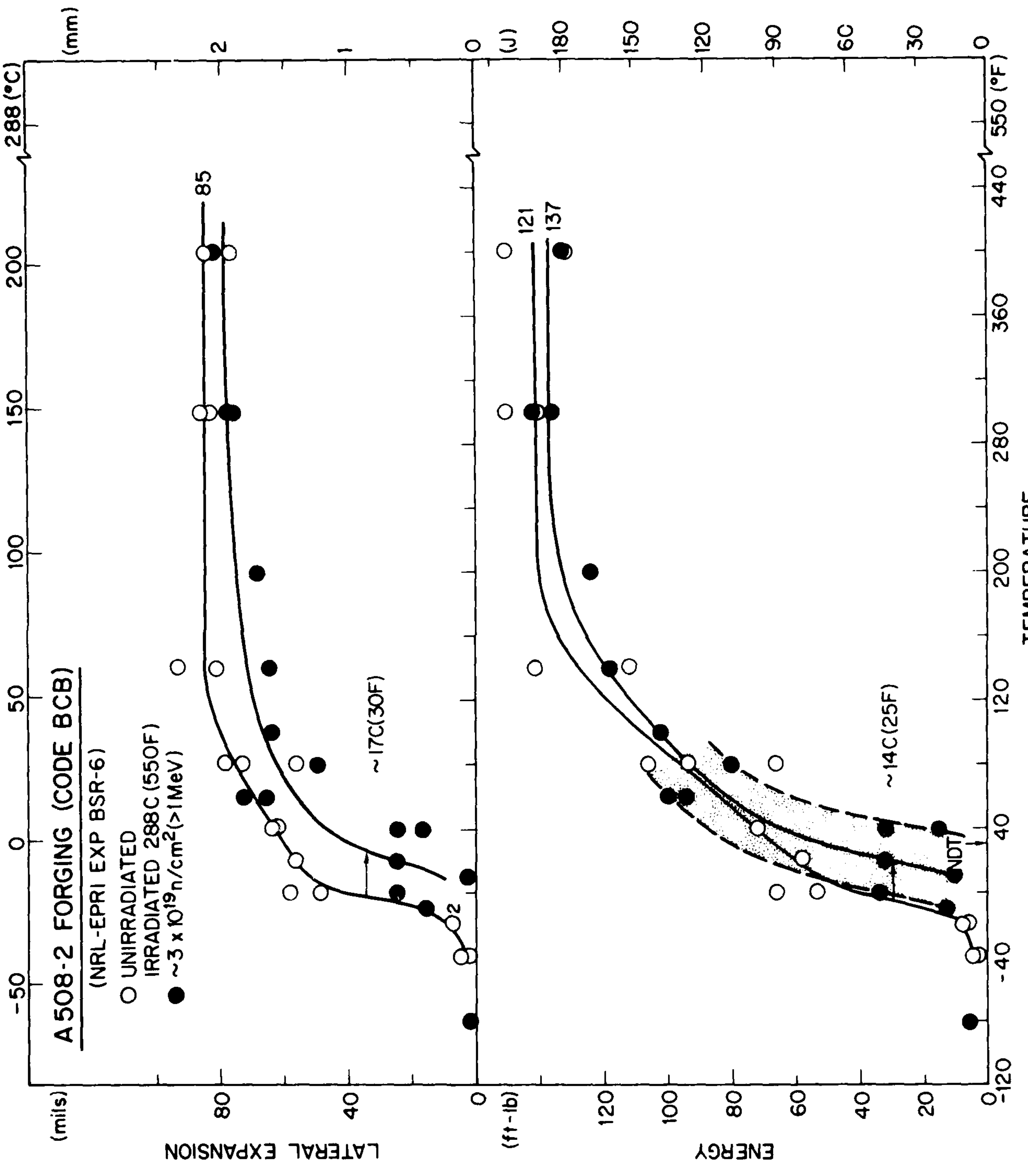

Fig. 9 - Charpy-V notch ductility of Forging BCB before and after 288°C irradiation. Irradiated specimens were contained in the same reactor experiment as the irradiated PCC_V specimens of Figure 8 [18].

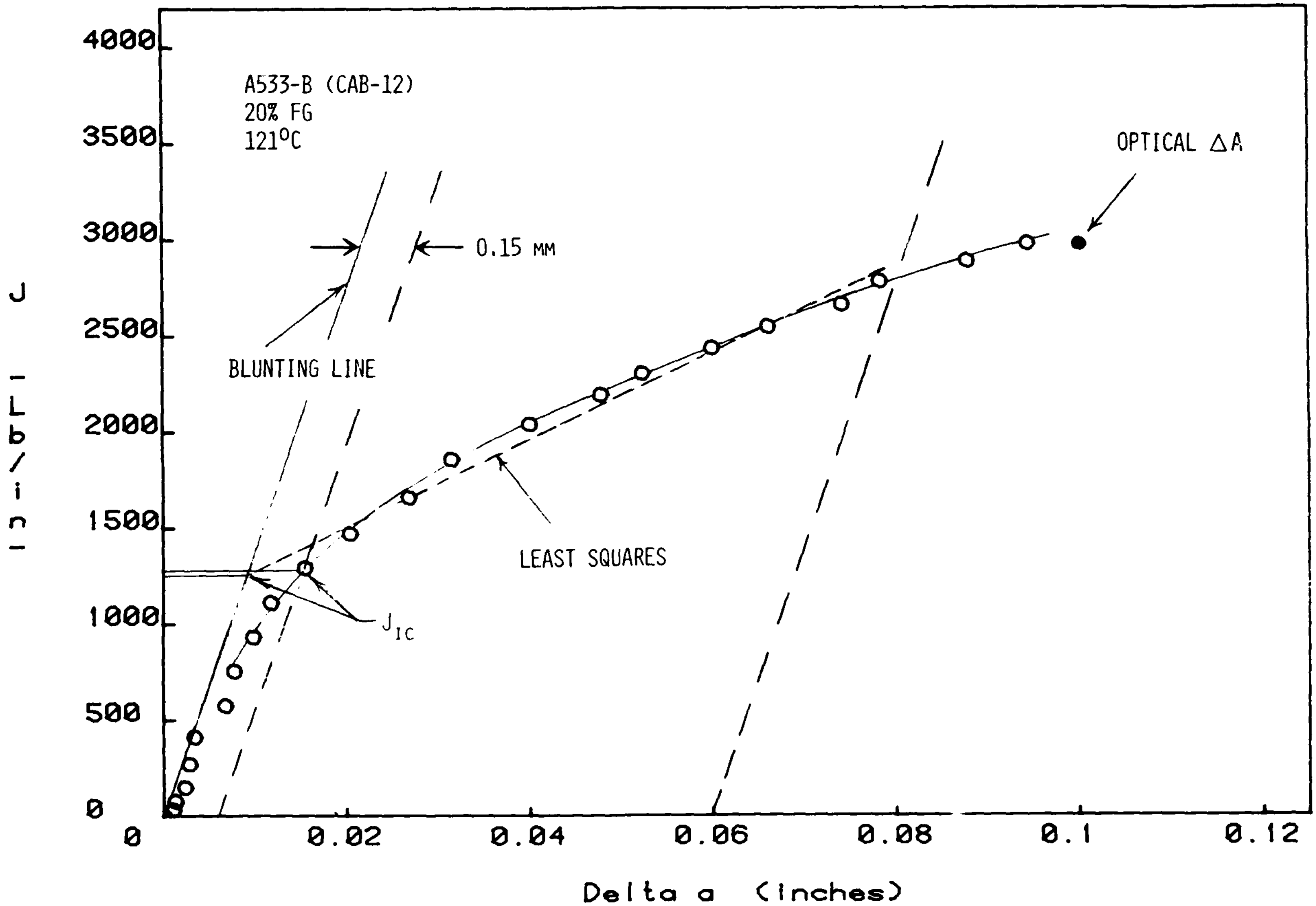

Fig. 10 - Typical R curve developed with a single CT specimen using the unloading compliance test method. The proposed ASTM definition of J_{Ic} based on a least squares fit of data beyond the blunting line is illustrated. An alternative definition of J_{Ic} proposed by NRL is based on the intersection of a smoothly drawn R curve and an exclusion line drawn 0.15 mm to the right of the blunting line [17,20].

$$K_{Jc} = \left(\frac{EJ_{Ic}}{1-\nu^2}\right)^{1/2}$$

where J_{Ic} is the initiation toughness, E is Young's modulus and ν is Poisson's ratio. The potential for crack instability is frequently being studied in terms of a tearing modulus concept advanced by Paris and others [19]. The tearing modulus, T, is defined as:

$$T = \left(\frac{E}{\sigma_f^2}\right)\left(\frac{dJ}{da}\right)$$

where σ_f is the flow stress and a is the crack length. Because of the power law relationship of the R-curve, an average value of the tearing modulus, T_{AVG}, is generally determined for the region between the dashed curves of Figure 10.

Experimental comparisons of C_v and CT test methods have also revealed similarities in their indications of irradiation effects. Specifically, the effects of irradiation and of irradiation and annealing on the K_{Jc} transition curve was found to correspond closely with the effects on the C_v transition curve measured at the 41J index. In terms of upper shelf performance trends however, significant differences have been observed. For example, in NRL IAR studies [14], essentially complete recovery in C_v energy level was found with 399°C annealing but only partial recovery in T_{AVG} values. Also, T_{AVG} shows an inverse relationship with temperature (Figure 11) whereas the C_v upper shelf energy of the material studied was essentially constant with temperature. The partial recovery in T_{AVG} was consistent with the flow stress trend but not with tensile ductility values. A correlation of the two test methods for a specific temperature has been possible however (Figure 12). Loss and co-workers evolved the correlation on the basis of eight nuclear vessel steels, with and without irradiation, and spanning the C_v energy range expected in service. The correlation has greatly enhanced the engineering signficance and usefulness of C_v data from reactor vessel surveillance. Additional details of the study are obtainable from references 14 and 20.

F. SUMMARY

USA studies of radiation embrittlement to reactor vessel materials have made significant progress in the last three years. Reported findings and newly developed data trends contribute on a broad front to the base technology. Demonstration tests which showed the worldwide range of applicability of new specifications and guidelines for tailoring steels for radiation resistance and correlation tests which compared notch ductility and fracture toughness changes with irradiation are illustrative of the range of studies conducted.

G. ACKNOWLEDGMENTS

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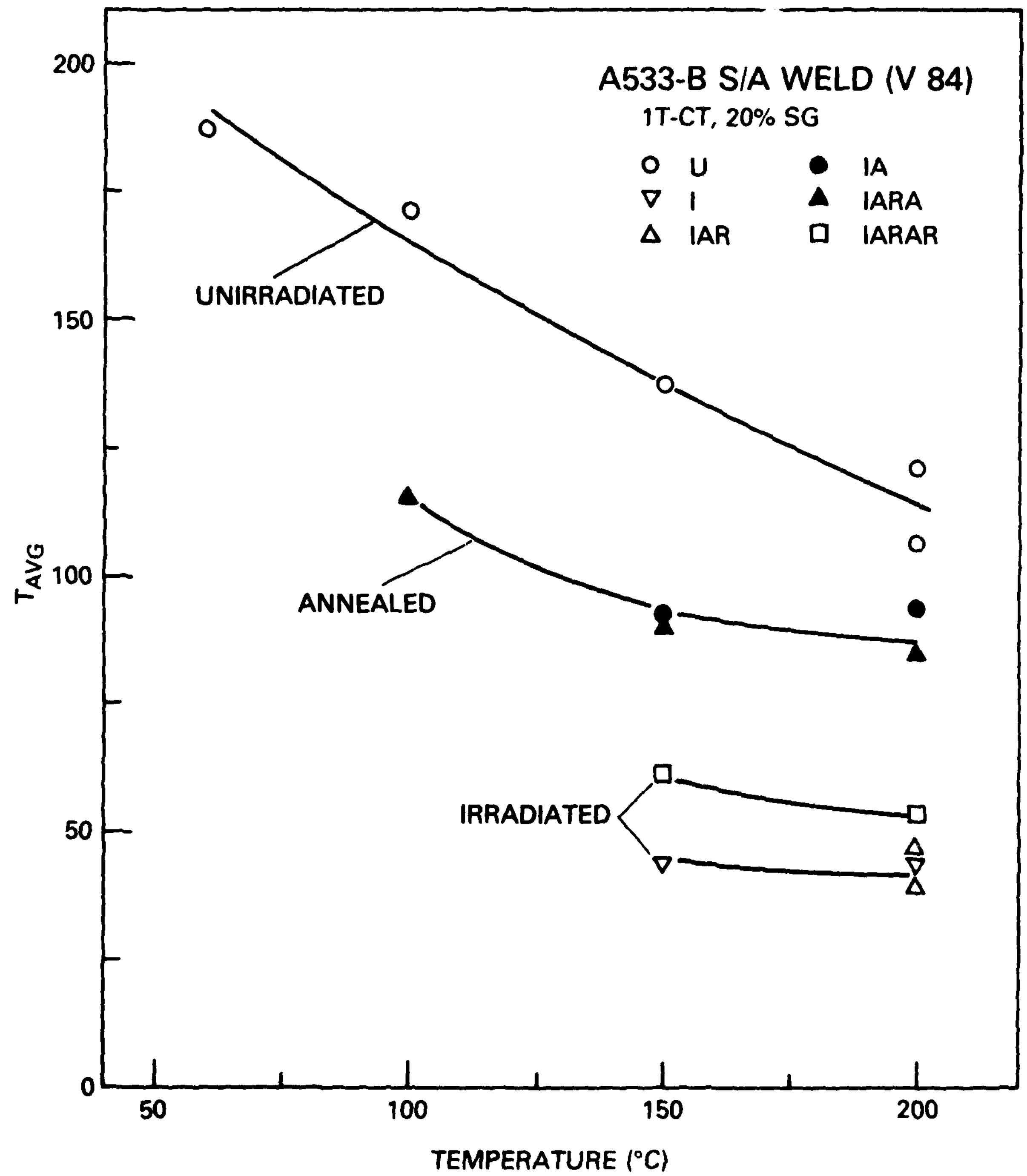

Fig. 11 - Variation of the average tearing modulus, T_{AVG}, with temperature for a submerged arc weld (0.35%Cu, 0.7%Ni) in the unirradiated, irradiated, annealed and annealed-and-reirradiated conditions [14].

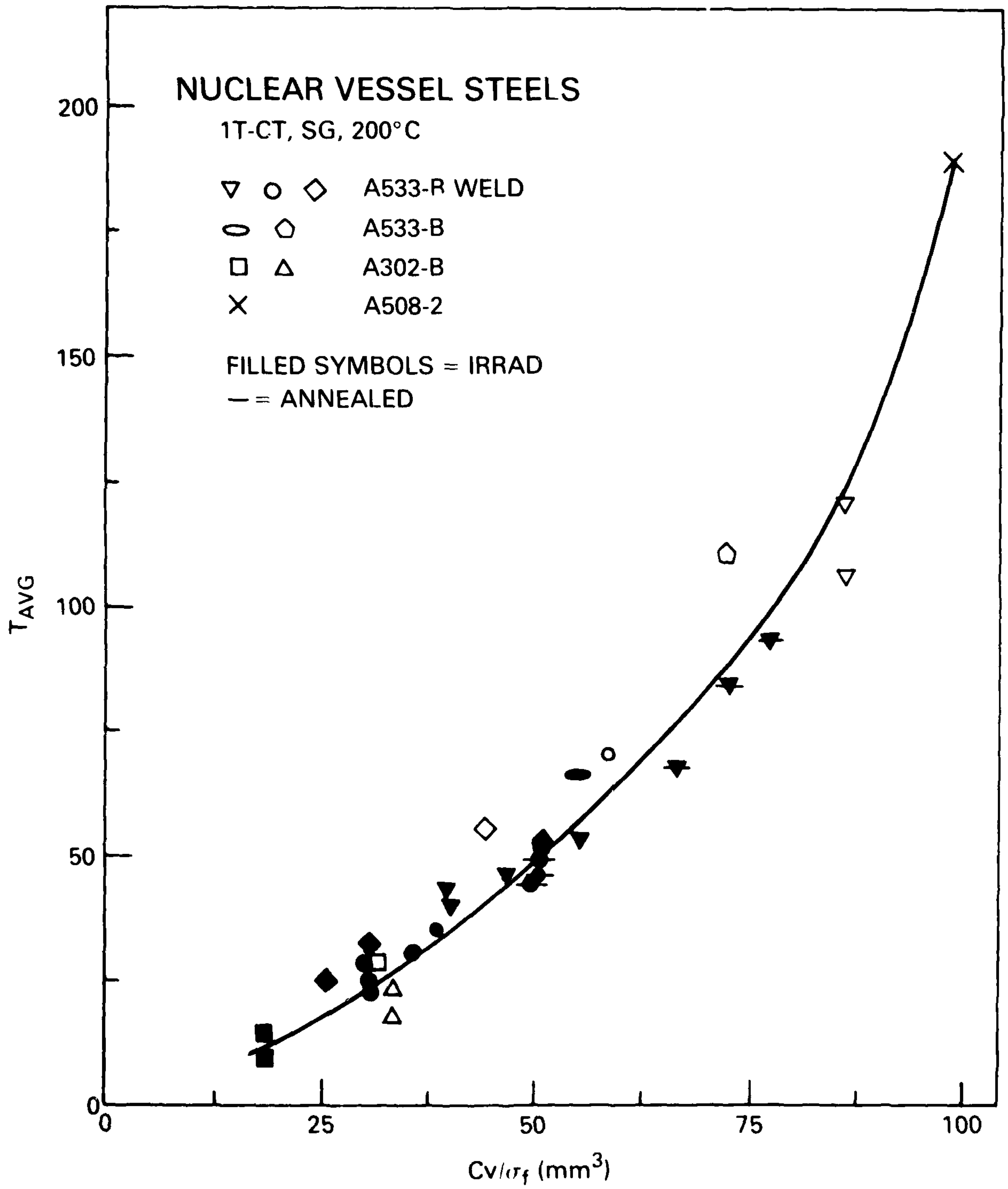

Fig. 12 - Correlation between C_v upper shelf energy and the average value of tearing modulus, T_{AVG}, for a crack extension less than 1.5 mm [14,20].

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16. ABSTRACT (200 words or less)

Advances by experimental research in the USA toward an improved understanding of property changes in steel by elevated temperature ($\sim 288^{\circ}C$) irradiation are summarized. Four areas of investigation are reviewed including the confirmation and demonstration of guidelines for radiation resistant steels, the isolation of metallurgical factors contributing to variable radiation embrittlement sensitivity, the qualification of in situ heat treatments for periodic vessel embrittlement relief, and the correlation of notch ductility and fracture toughness changes with irradiation.

Overall, the current state of the art provides both a high capability for tailoring steels for radiation service in new vessel construction and a promising method for controlling radiation embrittlement buildup in existing vessel construction.

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