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**EFFECTS OF ANNEALING TIME ON THE RECOVERY OF  
CHARPY V-NOTCH PROPERTIES OF IRRADIATED  
HIGH-COPPER WELD METAL\***

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## EFFECTS OF ANNEALING TIME ON THE RECOVERY OF CHARPY V-NOTCH PROPERTIES OF IRRADIATED HIGH-COPPER WELD METAL

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**REFERENCE:** Iskander, S. K., Sokolov, M. A., and Nanstad, R. K., "Effects of Annealing Time on the Recovery of Charpy V-Notch Properties of Irradiated High-Copper Weld Metal," *Effects of Radiation on Materials: 17th Volume, STP 1270*, David S. Gelles, Randy K. Nanstad, Arvind S. Kumar, and Edward A. Little, Editors, American Society for Testing and Materials, Philadelphia, 1996.

**ABSTRACT:** One of the options to mitigate the effects of irradiation on reactor pressure vessels is to thermally anneal them to restore the toughness properties that have been degraded by neutron irradiation. An important issue to be resolved is the effect on the toughness properties of reirradiating a vessel that has been annealed. This paper describes the annealing response of irradiated high-copper submerged-arc weld HSSI 73W. For this study, the weld has been annealed at 454°C (850°F) for lengths of time varying between 1 and 14 days. The Charpy V-notch 41-J (30-ft-lb) transition temperature ( $TT_{41J}$ ) almost fully recovered for the longest period studied, but recovered to a lesser degree for the shorter periods. No significant recovery of the  $TT_{41J}$  was observed for a 7-day anneal at 343°C (650°F). At 454°C for the durations studied, the values of the upper-shelf impact energy of irradiated and annealed weld metal exceeded the values in the unirradiated condition. Similar behavior was observed after aging the *unirradiated* weld metal at 460 and 490°C for 1 week.

**KEY WORDS:** annealing, irradiation, recovery, Charpy V-notch, transition temperature, upper-shelf energy, copper, weld metal, toughness, reactor pressure vessel

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### INTRODUCTION

Some early nuclear power reactor pressure vessels (RPVs) fabricated from certain types of steels may not meet the pertinent regulatory requirements as they near end of life. These regulatory requirements are promulgated in Appendix G of Title 10 of the *Code of Federal Regulations*, Part 50, "Fracture Toughness Requirements." It is believed that in

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the next decade or so, several vessels may exceed the limits set by the pressurized thermal shock reference temperature. In that case, thermal annealing may be needed to mitigate effects of neutron embrittlement on fracture toughness. A dozen or so RPVs have already been thermally annealed in Eastern Europe [1].

Historically, the toughness of RPVs has been monitored through surveillance programs in which predominantly Charpy V-notch (CVN) specimens are placed in capsules that are withdrawn and tested periodically. The degradation of fracture toughness is estimated from both the radiation-induced shift of the CVN 41-J (30-ft-lb) transition temperature ( $\Delta T_{41J}$ ) as well as the decrease in the upper-shelf energy (USE) level. The fracture toughness of irradiated RPV material is estimated from the American Society of Mechanical Engineers (ASME) *Boiler and Pressure Vessel Code* fracture toughness curves [2] by shifting them upward in temperature by  $\Delta T_{41J}$ . Recent advances in fracture mechanics allow the determination of the fracture toughness of the thick-section RPV steels from relatively small fracture toughness specimens, but for some of the earlier RPVs, the use of their CVN-based surveillance data and specimens will probably play a key role in the judgement of the efficacy of the anneal.

This paper discusses the effect of annealing on the CVN impact energy of irradiated high-copper welds. This research was performed within the Heavy Section Steel Irradiation (HSSI) Program sponsored by U.S. Nuclear Regulatory Commission at the Oak Ridge National Laboratory (ORNL). The long-term objectives of the HSSI Program annealing task are to determine the rates of fracture toughness degradation due to reembrittlement after annealing several critical irradiated materials and to relate them to the corresponding rates for unirradiated material upon irradiation, or to the recovery of the  $\Delta T_{41J}$  of irradiated and annealed material. The rate of reembrittlement is an important consideration since it will determine how long the plant may be safely operated after it is annealed. Most of the available data dealing with the recovery and reembrittlement response are based on the CVN impact energy, which must then be correlated with the fracture toughness.

## **DESCRIPTION OF MATERIAL USED**

The commercially fabricated submerged arc-weld used for this study, designated HSSI weld 73W, has been very extensively characterized in other HSSI tasks [3–7]. Large variations in the copper content (from 0.22 to 0.46%) have been observed in welds fabricated using weld wire with copper coating [8]. To avoid these large variations, the 73W weld wire was fabricated with copper added to the melt, consequently, the variation in copper content is very small. The chemical composition and standard deviation of HSSI Weld 73W, obtained from a large number of analyses, is shown in Table 1.

The CVN specimens used in this study were slightly smaller in one cross-sectional dimension than the standard full-size CVN specimens. This slightly smaller dimension of the specimens was dictated by the space available in the HSSI Fifth Irradiation Series capsules [9]. A typical arrangement of these specimens is shown in Fig. 1. The dimension

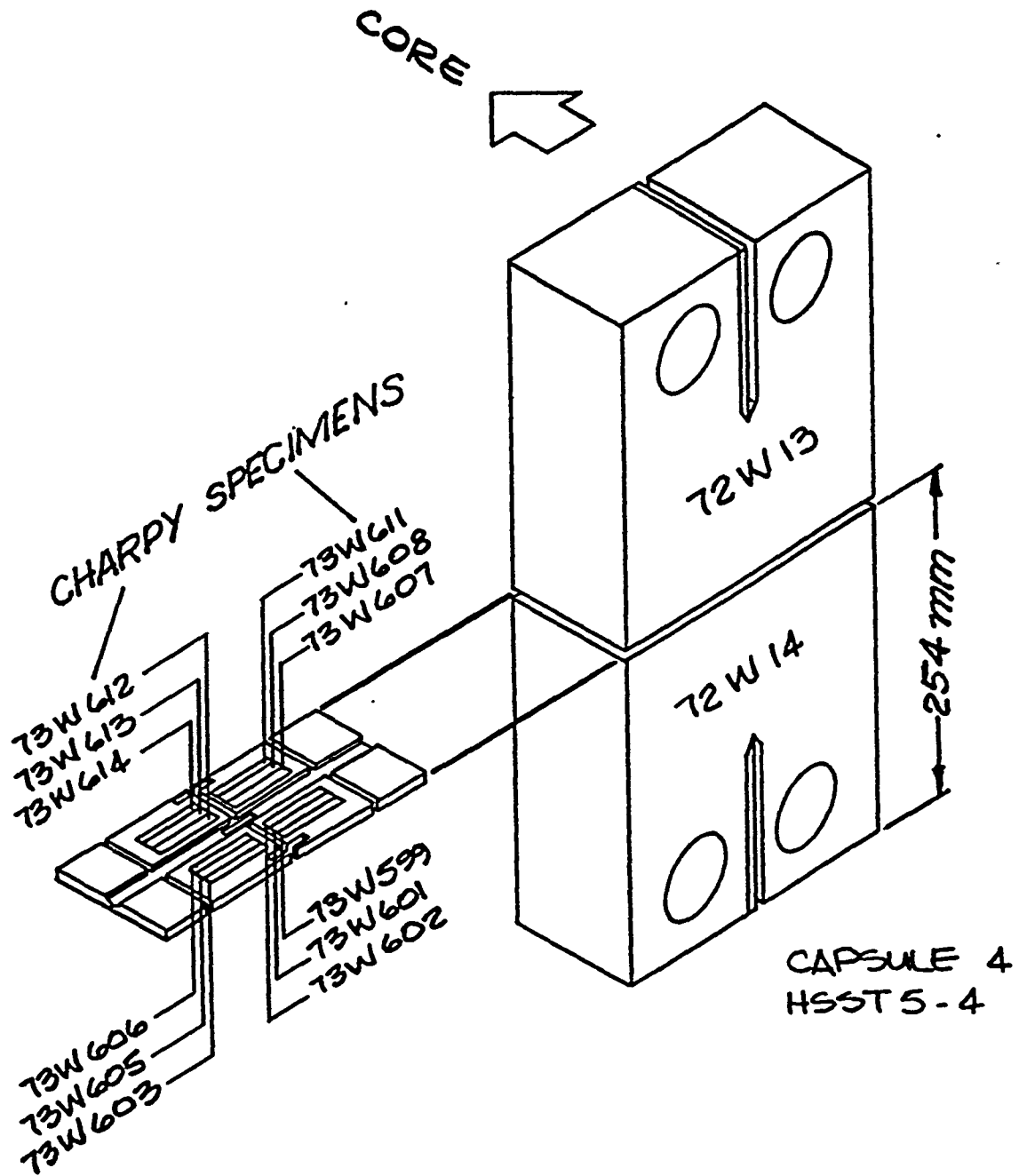


FIG. 1--The undersize Charpy V-notch specimens were irradiated in the available space of the Fifth Irradiation Series capsules.

normal to the notch of the "undersize" CVN specimens is 95% of full-size specimens, as shown in Fig. 2.

There were 120 undersized CVN specimens irradiated as part of the HSSI Fifth Irradiation Series, 12 in each of 10 capsules. They were irradiated at a nominal temperature of 288°C, and to average exposures of  $1.8 \times 10^{19}$  neutrons/cm<sup>2</sup> (> 1 MeV),  $1.1 \times 10^{20}$  neutrons/cm<sup>2</sup> (> 0.1 MeV), and 0.040 displacements per atom. The irradiation was performed in the Oak Ridge Research Reactor over a period of about 2 months, with an average flux of  $3.4 \times 10^{12}$  neutrons/(cm<sup>2</sup>-s) (> 1 MeV); full details are presented elsewhere [9].

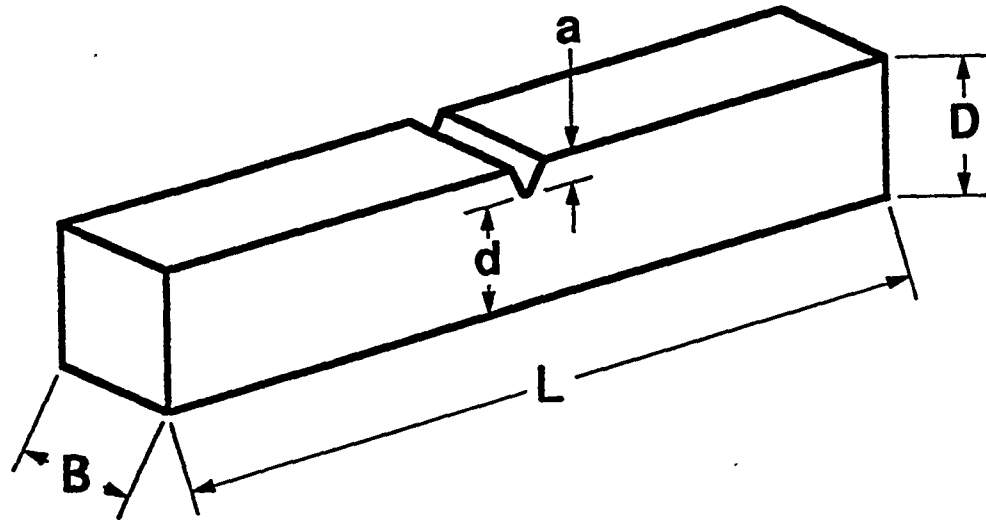
TABLE 1--Chemical composition and standard deviation of the various elements analyzed for HSSI weld 73W

Composition (wt % and standard deviation)									
C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V
0.098	1.56	0.005	0.005	0.45	0.25	0.60	0.58	0.31	0.003
0.007	0.026	0.0004	0.0006	0.028	0.006	0.006	0.009	0.010	0.0001

The results of CVN impact testing of unirradiated undersize CVN specimens are compared to those of full-size specimens in Fig. 3. Each of the curves shown is a hyperbolic tangent fit by nonlinear regression to the experimental results of testing 55 and 85 specimens for the undersize and full-size specimens, respectively. Although the slope of the curves in the transition region for the undersize specimens is somewhat steeper than for the full-size specimens, the CVN 41-J transition temperatures ( $TT_{41J}$ ) are approximately equal. As will be shown later, this difference in slope was not apparent in testing a smaller number of specimens (10 to 18) of either the irradiated or irradiated and annealed specimens of nominally the same geometry. Thus, this difference in slope may be due to the sensitivity of the undersize specimen to variations in the notch depth.

#### MATERIAL CONDITIONS INVESTIGATED

The irradiated CVN specimens were annealed at two temperatures, 343 and 454°C. These two temperatures have been often investigated as approximate lower and upper bounds of probable annealing temperatures [10]. The 343°C temperature could be used for a wet anneal. This is considerably simpler to perform than a dry anneal at 454°C since the reactor internals would not have to be removed. A 168-h annealing time was investigated for the lower temperature, and, since the recovery of the  $TT_{41J}$  was insignificant, no other annealing times were investigated. Four annealing times varying



	L (in) (mm)	B (in) (mm)	D (in) (mm)	d (in) (mm)	a (in) (mm)	a/D
Undersized specimens	2.165 55.00	0.394 10.00	0.375 9.52	0.303 7.71	0.072 1.82	0.191
Standard specimens	2.165 55.00	0.394 10.00	0.394 10.00	0.315 8.00	0.079 2.00	0.200

FIG. 2--Dimensions of the undersize Charpy V-notch specimens compared to the standard full-size specimens.

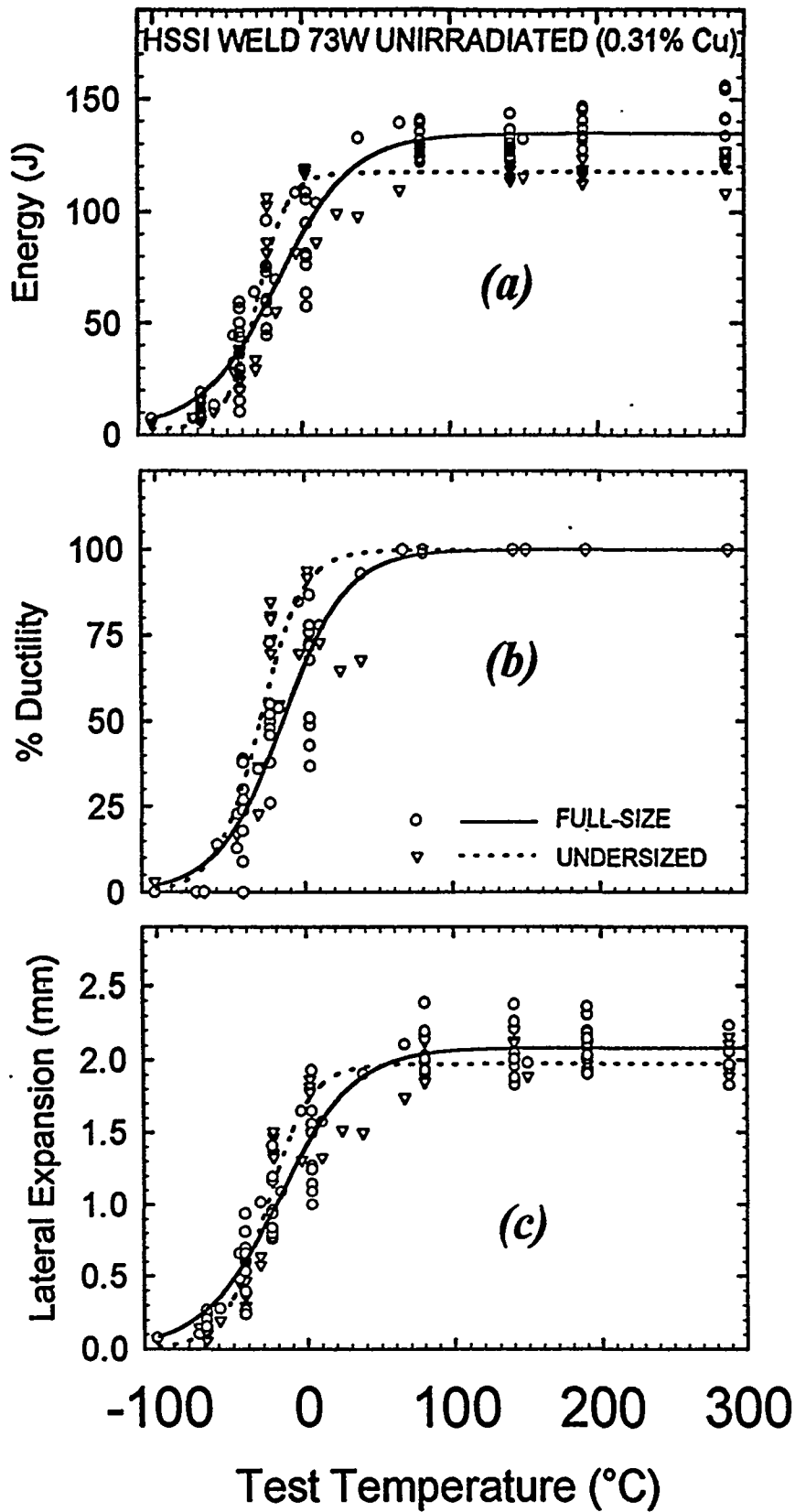


FIG. 3--Comparison of the Charpy V-notch impact energy of the unirradiated undersize HSSI weld 73W specimens to the full-size specimens: (a) energy, (b) ductile appearance, and (c) lateral expansion.

from 1 day to 2 weeks (336 h) were investigated at a temperature of 454°C. One of the results of this investigation is that annealing increased the USE to values greater than the unirradiated specimens. This was not unexpected since other investigators have also noted such an effect. Annealing, tempering and aging effects are diffusion processes where temperature is the dominant parameter and time is of secondary influence. As part of the original fabrication procedure, the 73W weld was postweld heat treated (PWHT) at a temperature of 607°C for 40 h, and thus exposure at the lower temperature of 454°C for 168 h would not be expected to have any effect. To determine whether this increase in USE was due to irradiation, unirradiated specimens were also aged at 460 and 490°C. The conditions investigated have been summarized in Table 2.

TABLE 2--Material conditions investigated

Material condition	Aging or annealing time (h)
Unirradiated	
Unirradiated and aged at 460°C (860°F)	168
Unirradiated and aged at 490°C (914°F)	168
Irradiated	
Irradiated and annealed 343°C(650°F)	168
Irradiated and annealed 454°C(850°F)	24, 96, 168, 336

## TEST RESULTS

The results of CVN tests on 73W welds for the conditions investigated are shown in Figs. 4 through 8. The recovery of CVN impact properties is measured by the changes in values of the USE and  $TT_{41J}$  before and after annealing when compared to the unirradiated values. The values of the USE and  $TT_{41J}$  were calculated from a nonlinear regression fit of a hyperbolic tangent equation to the CVN impact energy results. The hyperbolic tangent equation was also fitted to the experimental values of percent ductile shear appearance and lateral expansion, and is of the form:

$$y = \frac{US + LS}{2} + \frac{US - LS}{2} \operatorname{Tanh} \left( \frac{T - MTT}{TZW/2} \right), \quad (1)$$

where

- y = energy, percent ductile shear appearance, or lateral expansion
- US and LS = upper- and lower-shelf values, respectively,
- T = test temperature,
- MTT = mid-transition temperature, and,
- TZW = transition zone width.



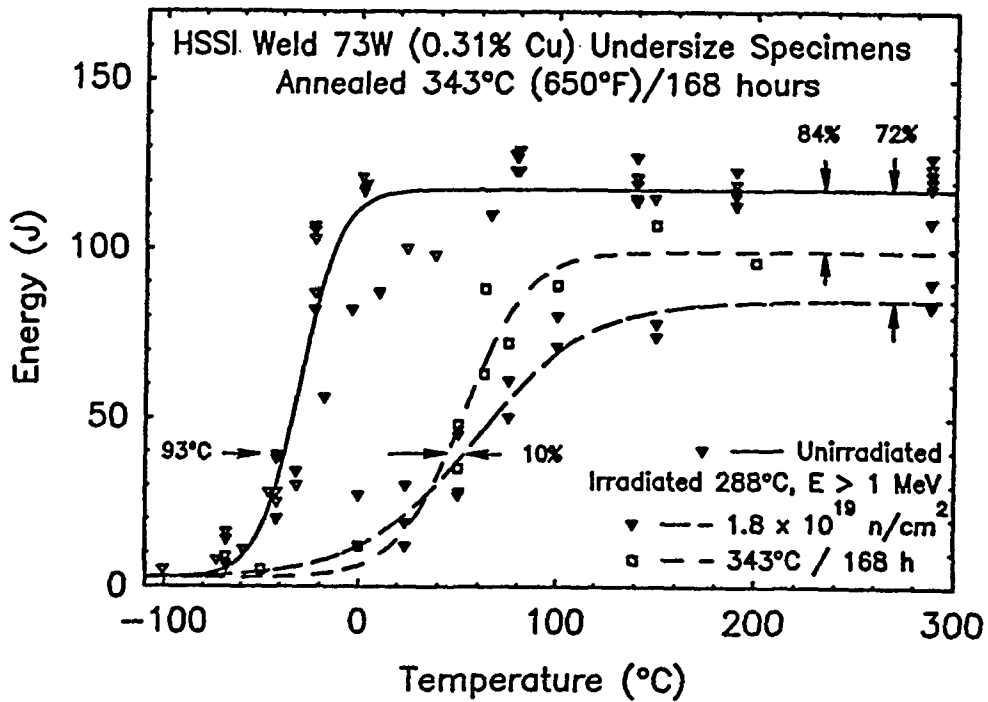


FIG. 4--Results of testing undersize irradiated Charpy V-notch specimens annealed at 343°C for 168 h compared to specimens in the unirradiated and irradiated conditions. The percent recovery is also indicated.

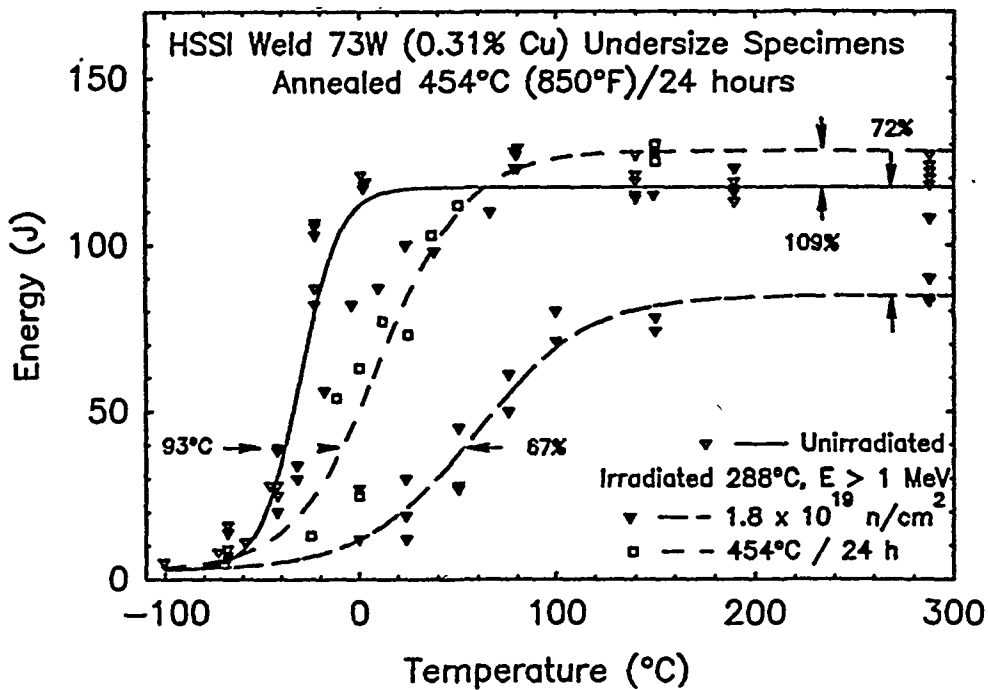


FIG. 5--Results of testing undersize irradiated Charpy V-notch specimens annealed at 454°C for 24 h compared to specimens in the unirradiated and irradiated conditions. The percent recovery is also indicated.

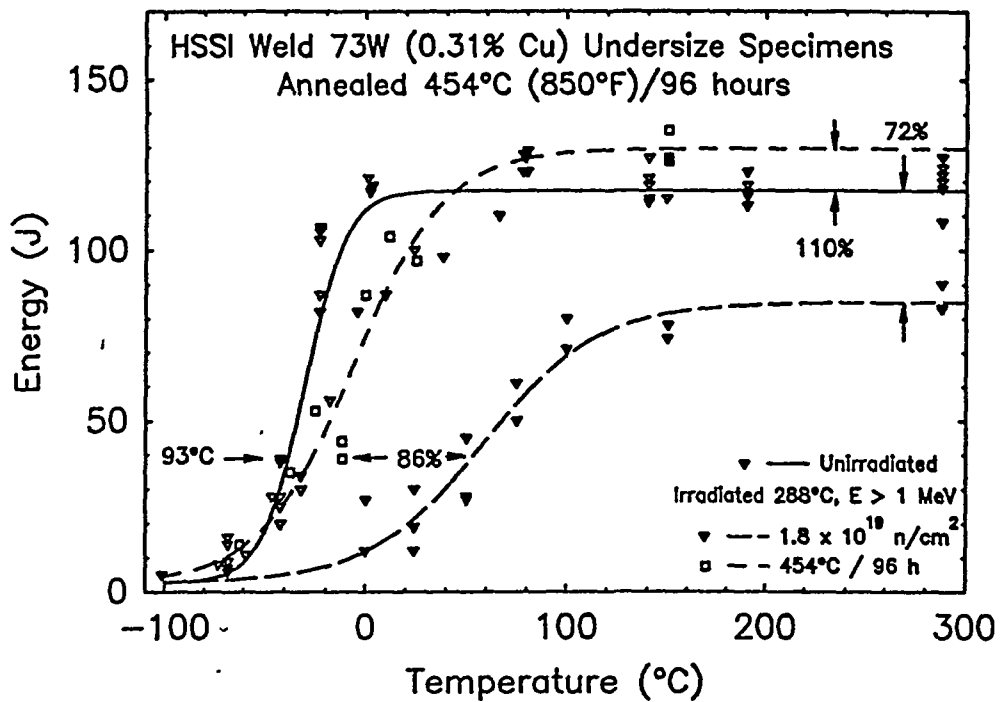


FIG. 6--Results of testing undersize irradiated Charpy V-notch specimens annealed at 454°C for 96 h compared to specimens in the unirradiated and irradiated conditions. The percent recovery is also indicated.

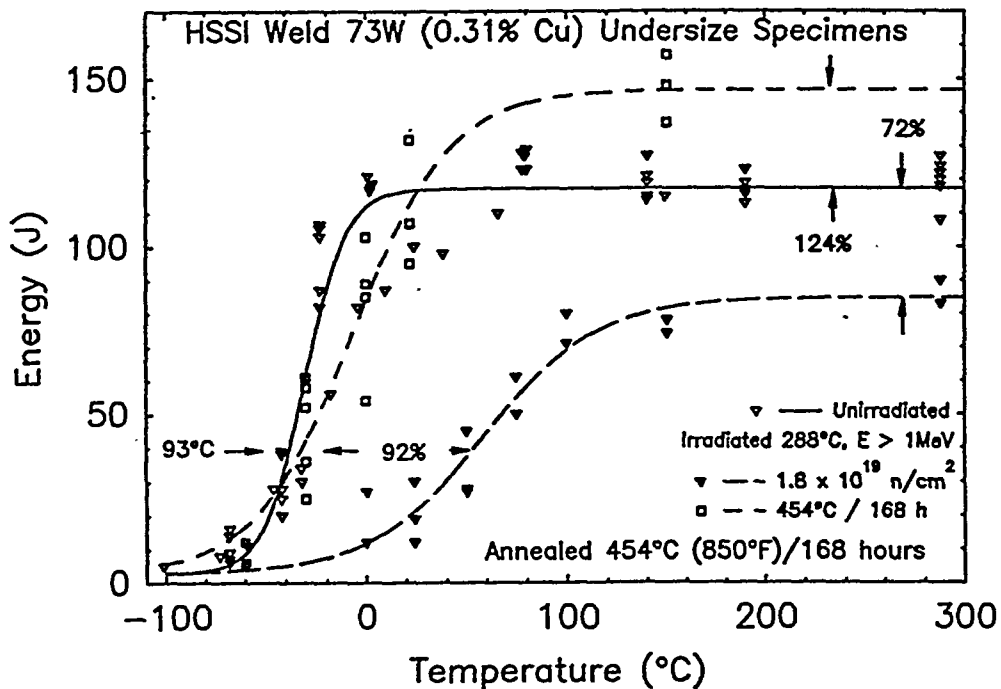


FIG. 7--Results of testing undersize irradiated Charpy V-notch specimens annealed at 454°C for 168 h compared to specimens in the unirradiated and irradiated conditions. The percent recovery is also indicated.

In the case of fitting energy values, the LS was prescribed to be 2.7 J, the average value obtained by testing five submerged-arc welds at liquid nitrogen temperature of  $-196^{\circ}\text{C}$  [8]. For lateral expansion and percent ductile shear appearance, the value of LS was prescribed to be zero. The upper-shelf value, US, of ductile shear was prescribed to be 100%. Except for these prescribed values, US, MTT, and TZW were fitting parameters.

The percent recovery of the  $TT_{41J}$  is defined as the ratio of the residual transition temperature shift after annealing to the shift due to irradiation,  $\Delta TT_{41J}$ , or:

$$\% \text{ Recovery } TT_{41J} = \frac{[TT_{41J}]_{irr} - [TT_{41J}]_{ann}}{[TT_{41J}]_{irr} - [TT_{41J}]_{unirr}} \cdot 100, \quad (2)$$

where  $TT_{41J}$  is the transition temperature at the 41-J energy level for the condition indicated by the outer subscript. Thus, the percent recovery is related to the shift,  $\Delta TT_{41J}$ , due to irradiation.

The percent recovery of the USE could also be defined in an analogous manner to Eq. (2):

$$\% \text{ USE Recovery} = \frac{USE_{ann} - USE_{irr}}{USE_{unirr} - USE_{irr}} \cdot 100. \quad (3)$$

On the other hand, changes in USE could be related to the unirradiated value, hence the percent recovery of the USE may also be defined as the ratio of the values of the USE in the irradiated and annealed (or aged) condition to the unirradiated condition:

$$\% \text{ USE Recovery} = \frac{USE_{ann}}{USE_{unirr}} \cdot 100. \quad (4)$$

The disadvantage of Eq. (4) is that it makes no distinction in the case in which there is no recovery, indicating instead the loss of USE due to irradiation. Its main advantage is that, in cases of USE recovery (which is usually the case), the value relates to the unirradiated value. The disadvantage of Eq. (3) is that it cannot be applied to the case of unirradiated and aged material since there is no "irradiated" condition, while Eq. (4) allows comparisons between the changes of the USE in the unirradiated/aged and irradiated/annealed conditions. In this paper, both definitions of the percent recovery of USE have been used.

Except for the  $343^{\circ}\text{C}$  annealing temperature, which did not show any significant recovery in the  $TT_{41J}$  as mentioned previously, the degree of CVN impact energy recovery was dependent on the length of annealing time. The  $TT_{41J}$ , USE, change, and percentage

recovery for each of the conditions investigated has been summarized in Table 3. The percentage recovery of both the  $TT_{411}$  and the USE have been plotted in Fig. 9 for annealing at 454°C.

## DISCUSSION

Annealing the HSSI weld 73W at 454°C for various lengths of time from 24 to 336 h has "over-recovered" the USE to values that exceeded the unirradiated ones, which has been also been observed in other investigations. As mentioned above, this increase cannot be explained by a simple time-temperature diffusion process in which temperature is the dominant parameter, since the weld was already PWHT at the higher temperature of 607°C. Thus, the annealing at 454°C would not be expected to produce such changes in impact energy. Aging of unirradiated HSSI weld 73W at 460 and 490°C for 168 h also increased the value of USE, so this increase could not be explained solely by changes in the material caused by irradiation. The recovery by annealing of the neutron irradiation-induced transition temperature shift can be understood, to a large degree, in terms of coarsening or over aging and decrease in the number density of the copper-rich precipitates that are believed to be the primary cause of the transition temperature shift [11]. However, the reasons for the over-recovery of the USE in the unirradiated and irradiated conditions is still unknown, and the HSSI program will continue to investigate this phenomenon. It should be noted that the aging of unirradiated HSSI weld 73W did not cause any noticeable changes in the value of  $TT_{411}$ .

Annealing HSSI weld 73W at 454°C for 24 h recovered about two-thirds of the transition temperature shift caused by the neutron irradiation. Annealing at this temperature for longer times increases the recovery, but at a decreasing rate, so that doubling the annealing time from 168 to 336 h increases the percent recovery from 92 to 96%, which may not be a reasonable return for the extra annealing time invested.

It is significant that in all the above cases of annealing of irradiated HSSI weld 73W at 454°C, the percentage recovery of the USE was greater than the percentage recovery of  $TT_{411}$ . This was also observed in other investigations as can be shown by plotting the percent recovery of upper shelf versus the percent recovery of the  $TT_{411}$  for RPV steels. The data in Fig. 10 of the % USE Recovery, Eq. (3), versus the % Recovery  $TT_{411}$ , Eq. (2), show that the majority of the data lie above the straight line with a 1:1 slope. The data in Fig. 10 were extracted from the ORNL test reactor data base [12]. This confirms the results from this investigation, namely that (1) the USE recovers faster than the  $TT_{411}$ , and (2) the percentage recovery of USE, as defined by either Eqs. (3) or (4), is greater than 100%. This result can be useful in cases where the number of specimens available is less than desired for an adequate determination of both  $TT_{411}$  and USE. More specimens could be devoted to determine the  $TT_{411}$ , and fewer specimens could be used to confirm the recovery of the USE.

TABLE 3--Values of the 41-J transition temperature and upper-shelf energy for all conditions investigated  
(blank values are unavailable or not applicable)

Condition	Annealing/aging		41-J transition temperature			Upper-shelf energy			
	Temperature (°C)	Time (h)	Value (°C)	Residual shift <sup>a</sup> (K)	Recovery <sup>b</sup> (%)	Value (J)	Change <sup>c</sup> (J)	Change <sup>d</sup> (%)	Change <sup>e</sup> (%)
Unirradiated			-38			118			
Irradiated			55	93		85	-33		72
Irradiated and annealed	343	168	46	84	10	99	-19	42	84
Irradiated and annealed	454	24	-7	31	67	128	10	130	108
Irradiated and annealed	454	96	-25	13	86	130	12	136	110
Irradiated and annealed	454	168	-31	7	92	147	29	188	125
Irradiated and annealed	454	336	-34	4	96	154	36	209	131
Unirradiated and aged	460	168				138	20		117
Unirradiated and aged	490	168				138	20		117

<sup>a</sup>Change from unirradiated value, positive values indicated an increase in the transition temperature.  
<sup>b</sup>Defined by Eq. (2).  
<sup>c</sup>Change from unirradiated value, positive values indicate an increase in upper shelf.  
<sup>d</sup>Defined by Eq. (3).  
<sup>e</sup>Defined by Eq. (4).

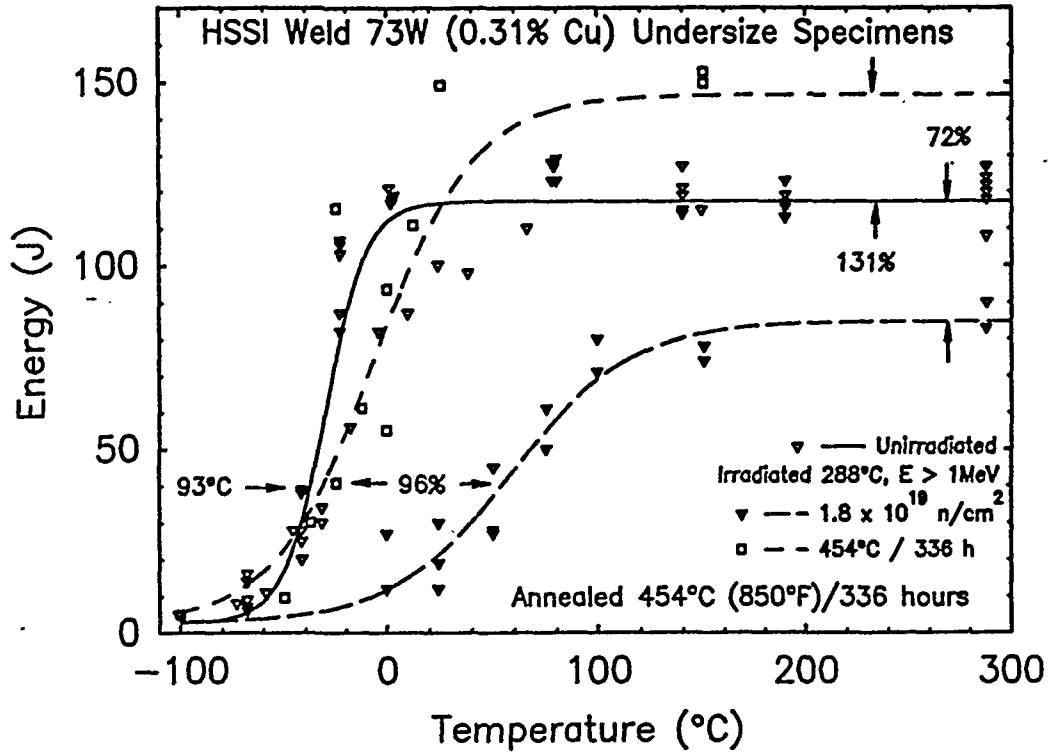


FIG. 8--Results of testing undersize irradiated Charpy V-notch specimens annealed at 454°C for 336 h compared to specimens in the unirradiated and irradiated conditions. The percent recovery is also indicated.

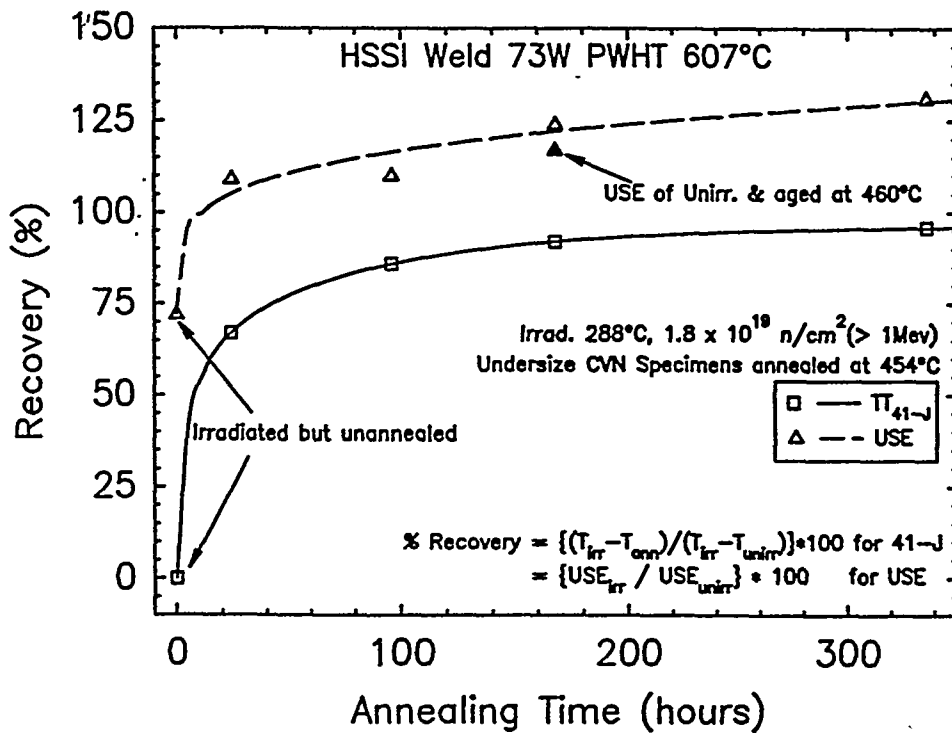


FIG. 9--Summary of percent recovery of the 41-J energy level transition temperature and the upper-shelf energy.

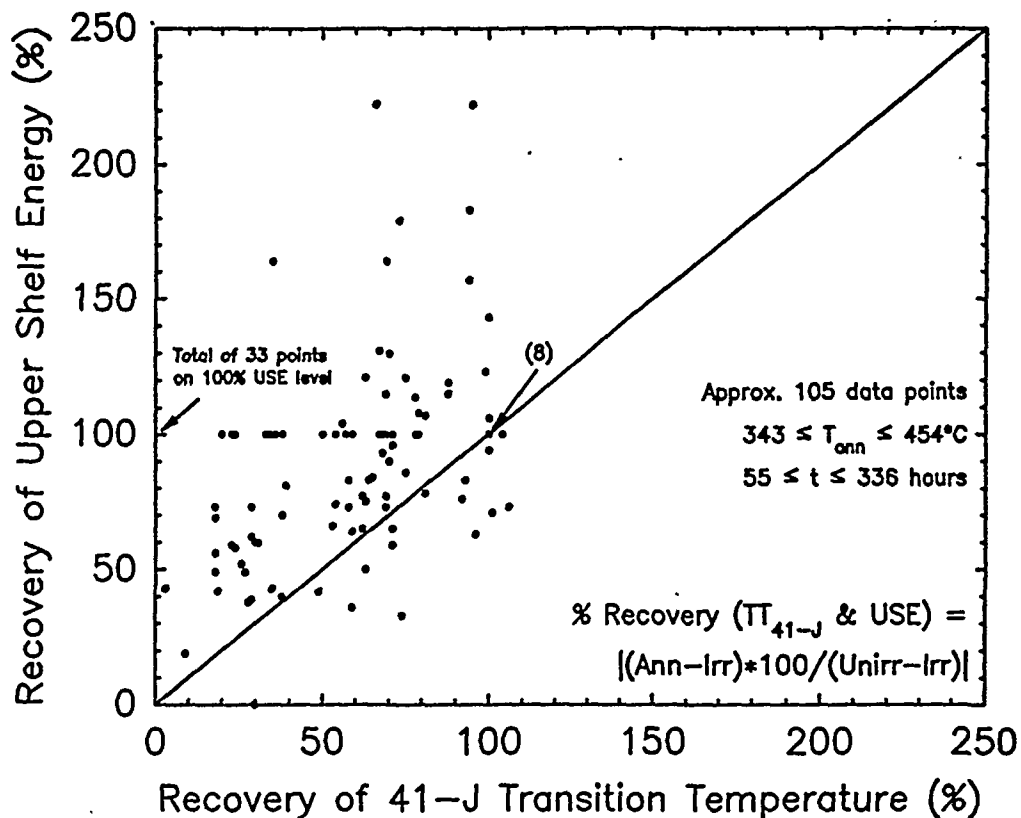


FIG. 10--Percent recovery of the upper-shelf energy versus percent recovery of the 41-J energy level transition temperature shift.

The rate of toughness degradation of irradiated and annealed RPV steels upon reirradiation is the present focus of investigations at ORNL. This is a major consideration in determining how long a RPV could be operated safely after it is annealed. It is possible that for some of the older RPVs for which annealing will be considered, no archival material is available, and the surveillance data from CVN specimens are the only data available that describe the rate of embrittlement. Thus, relating the rate of toughness degradation due to reirradiation to the rate of transition temperature shift of CVN specimens (the so-called "trend curve") from the unirradiated state becomes important.

## CONCLUSIONS

HSSI weld 73W was annealed at two temperatures for various lengths of time. The following conclusions could be deduced from the results:

1. Annealing of HSSI weld 73W has resulted in various degrees of recovery of the transition temperature and USE that depend strongly upon the annealing temperature and to a somewhat lesser degree upon the annealing time.
2. Annealing at  $454^{\circ}\text{C}$  for 24 h recovered about two-thirds of the transition temperature shift caused by the neutron irradiation.

3. Annealing at 454°C for longer times increases the recovery, but at a decreasing rate, so that doubling the annealing time from 168 to 336 h increases the recovery from 92 to 96%, which may not be a reasonable return for the extra annealing time invested.

4. Annealing at 343°C has resulted in an insignificant recovery of the transition temperature and a relatively small recovery in the USE.

5. Annealing at 454°C for various lengths of time from 24 to 336 h has "over-recovered" the USE to values that exceeded the unirradiated values.

6. Over-recovery of the USE was also observed after aging the *unirradiated* weld at 460 and 490°C for 1 week, and thus over-recovery is not solely associated with recovery of irradiation-induced damage.

The reasons for the over-recovery of the USE in the unirradiated and irradiated conditions are still not understood, particularly since HSSI weld 73W has been postweld heat treated at 607°C for 40 h. The HSSI Program will continue to investigate this phenomenon.

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