A study of the mechanical property changes of irradiation embrittled pressure vessel steels and their response to annealing treatments

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Isochronal and isothermal heat treatments have been used to study the recovery of hardness of an irradiated pressure vessel steel forging for purposes of planning and realizing IAR (Irradiated-Annealed-Reirradiated) experiments. Charpy V notch tests have been performed to assess the toughness of the material irradiated to various fluences up to a maximum of $5x10^{19}$ n/cm^2, E > 1MeV at 290°C with and without an intermediate annealing treatment at 450Cx168h. The effect of the intermediate annealing was evident. The recovery of the upper shelf energies was strongly enhanced by a thermal ageing effect due to the annealing treatment for all fluence levels investigated compared to the irradiated condition. The transition temperature shifts exhibited a less straight foreward behaviour due to the mentioned ageing effect which opposed the recovery process for this property leading to a net shift increase at lower and to a net recovery benefit at higher fluence levels. A phenomenological model description for the IAR embrittlement-recovery path is suggested. For this material and these irradiation conditions a plant life extension (PLEX) may be brought about if a specific annealing treatment is applied at a fluence level that is half the anticipated target fluence F for PLEX. In this case it was found that F > $1.6x10^{19}$ n/cm^2.

1 Introduction

The response of reactor pressure vessel (RPV) steels to neutron irradiation embrittlement effects has been studied internationally for many years (e.g. Steele 1983, 1986). Phenomenologically it has become clear that many factors dictate a steel's propensity to neutron embrittlement and these include the irradiation conditions, chemical composition and microstructure. The role of copper as an impurity element in aggrevating embrittlement became evident early on in the investigations (Hawthorne 1979) using Charpy V notch (Cv) testing. At the present time, world wide programmes are being carried out (IAEA 1986) to investigate the precise nature of the embrittlement using both mechanical and microphysical methods (Beaven et al. 1986)

One way to alleviate irradiation induced embrittlement is to perform annealing heat treatments on the material whereby the Charpy upper shelf energy (USE) and transition temperature (TT) may be restored to their approximate original values (Hawthorne 1983). It has been demonstrated that the annealing temperature must be at least 100 to 150C higher than the irradiation temperature (290C) if a substantial recovery is to be achieved in treatment times of up to 168h (Hawthorne 1979, Johnson 1982, Klausnitzer 1979). Two main aspects are of interest using annealing trials namely:

i) studies of the recovery processes using the annealing temperature as an experimental parameter coupled with microphysical measurements (Ghazi-Wakili et al 1987), and

ii) the practical in-situ annealing of RPV's to remove the radiation damage and to restore the mechanical properties.

The latter aspect is of great technological and economic importance since a safe PLEX (plant life extension) may be realisable (Hawthorne 1985).

Using the Cv notch energy as an indicator, the amount of effective radiation embrittlement recovery can be determined for a given annealing treatment in a series of IAR experiments. The dominant mechanisms responsible for the effective recovery of ΔTT and ΔUSE is the question of our main concern in this paper. Hawthorne started IAR investigations some years ago (Hawthorne 1978) and some results concerning the recovery and reembrittlement path (for the work cited an important question) are becoming available (Hawthorne 1985). IAR experiments are also part of our extended research programme on neutron embrittlement phenomena in RPV steels, including various mechanical and microphysical experimental techniques (Solt et al, this conference, section G). This paper presents the first results of such an IAR study.

Section 2 presents the standard mechanical behaviour of the investigated forging with respect to the Vickers microhardness as a function of annealing treatments and with respect to Cv toughness as a function of irradiation conditions. These results formed the basis for the planning of the IAR experiments. Section 3 describes the subsequent Cv tests and, based on their fluence dependence it was possible to describe a phenomenological model for the IAR embrittlement path. In the last section, first conclusions are drawn concerning PLEX which may be achieved by annealing and we mention further steps that are underway to complete the present study.

2 Standard Mechanical Properties – Response to Neutron Irradiation and Annealing

In this section, a study of the microhardness and its development with irradiation and subsequent annealing as well as the characteristic response of the Charpy toughness to neutron irradiation is presented.

The Vickers microhardness (HV0.2) using a load of 1.962N for 15s was chosen as a quick and simple indicator for the annealing response. Both isochronal and isothermal experiments were performed in order to determine the temperature and time where a substantial recovery in hardness would occur. These parameters were used to select a practical annealing schedule for the IAR experiments (see section 3).

The standard Charpy test was used as an indicator of toughness. Increases in the TT's indexed to the 41J energy level and irradiation induced changes in the USE's were related to the unirradiated reference condition. Such results, depending on the irradiation exposure level (target fluences are indicated in Table 1) served as a reference when studying the annealing and reirradiation behaviour of the Cv toughness in section 3.

The forging used was a fully qualified mock-up of a commercial nuclear PV steel. The chemical composition, heat treatment and mechanical properties were all certified and accepted by the utility owners and the nuclear regulatory authority as being representative for the PV in question. The chemical composition, heat treatment schedule and the as-received mechanical properties are given in Table 1.

The specimens were loaded into a fully instrumented irradiation rig (thermocouples and neutron monitors) and were kept under a protective gas mixture of helium and nitrogen forming part of a loop. Further irradiation conditions are summarized in Table 1. The irradiation temperature of 290C was achieved by using gamma heating in the specimens (specimen volume = 30 x 55 x 370 mm^3), a specific gas gap geometry between the elongated-cylindrically shaped specimen holder and the irradiation rig and a gas mixture control. The neutron spectrum was tailored to represent that of a particular commercial PWR at the inside surface of the PV.

Table 1: Type of investigated RPV steel, composition, as-received properties, irradiation conditions and tests performed. Only alloy and impurity elements of interest are shown.

Forging 270 mm thickness[a] Code 1.2 MD 07 B									
Composition					Mechanical properties				
Si	Mn	Ni	P	Cu	Rp0.2	Rm	HB	DBTT(41J)	USE
		[wt %]			[N/mm^2]			[°C]	[J]
0.29	1.47	0.87	0.02	0.17	483	635	207	-37	150
Irradiation conditions[b]					Testing[c] at target fluences[d]				
T_{irr}		Spectrum	Flux ϕ_{CB}		HV0.2		Cv		
[°C]		[E >1MeV]	[ϕ_{PWR}]				[10^{19} n/cm^2]		
290±Δ		PWR tailored	100		1.9	0.5	1.7	3.0	5.0

a Heat treatment: to 875C in 7h, hold 10h, water quench, temper to 635C in 6h, hold 11h, furnace cool to 300C, air cool.

b Δ = 5C in 80% of irradiation time and Δ = 10C in 20% of irradiation time; temperature control by thermocouples located at strategic points scattered over the specimen volume. ϕ_{PWR} = flux level typical of a PWR (~5 x $10^{10}cm^{-2}s^{-1}$), ϕ_{CB} = flux level at the core edge positions of the pool type 10 MW materials test reactor SAPHIR.

c Standard Charpy ISO-V-specimens, notch direction T–L; average taken of ≥ 5 HV determinations.

d Neutron fluence determination: Fe, Cu, Ni, Nb.

2.1 Microhardness (HV0.2)

The hardness in the unirradiated condition was 234±7, in the irradiated condition it was 293±9. The isochronal behaviour of the forging irradiated to $1.9x10^{19}n/cm^2$, at 290C is shown in Figure 1. It can be seen that the microhardness began to recover at ~ 410C with a holding time of 1h. The maximum rate of recovery appeared between 440C and 460C as indicated by the derivative. Consequently an annealing temperature of 450C was chosen

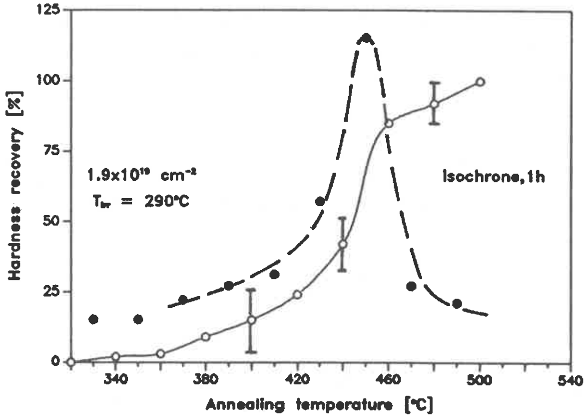

Figure 1 Isochronal behaviour of the forging. Appreciable recovery of the normalised hardness occurred at temperatures > 410C. The maximum rate of recovery (indicated by the dashed derivative curve) appeared at 450 C; this temperature was selected for the IAR experiments.

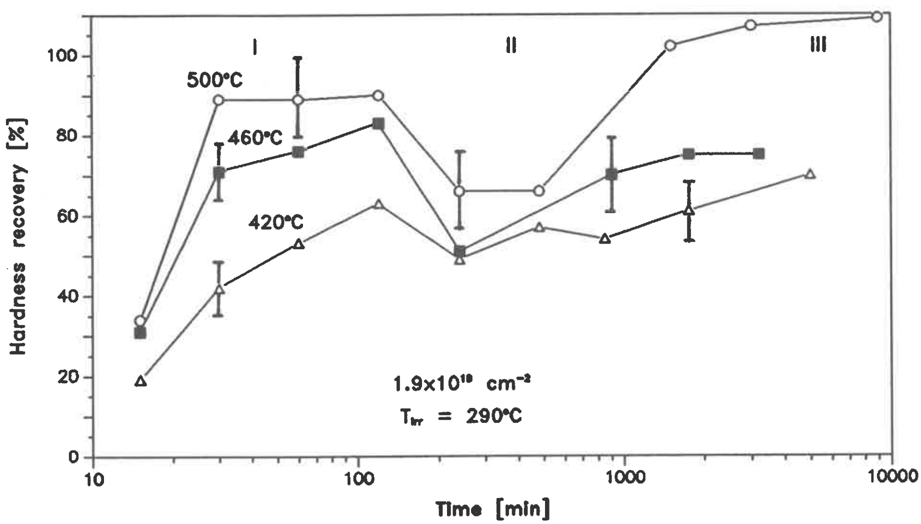

Figure 2 Isothermal behaviour of the forging. The curves indicate the three regions associated with the rapid recovery of the hardness (I), a loss of recovery (II) and resoftening (III). At least 80% recovery in the normalised hardness is expected to have occurred after 168h x 450 C. Error bars correspond to estimates of standard deviations. For any data point the average was taken of at least 5 HV determinations.

for the IAR experiments. Positron annhilation experiments have also shown an annealing stage at 450C (Ghazi-Wakili et al 1987).

The isothermal behaviour is depicted in Figure 2. Three main regions, denoted as I, II and III could be separated from the measured curves. Region I was characterized by a rapid recovery in microhardness. At 420C at least 50% recovery was present after 1h whilst $\sim$ 90% recovery was obtained at 500C for this time. Region II was characterized by a loss in recovery, i.e a rehardening took place. The effect may be due to a form of radiation anneal hardening or ageing or both (Pachur 1982, Mager 1978). As heating continued, the recovery in hardness commenced again (region III) and reached a maximum value depending on the holding temperature. Annealing times of up to 168h were used and this time was also selected for the IAR experiment (see section 3.1). It is of interest to note that at least 80% recovery in hardness would be achieved using 450 C x 168h as annealing conditions and this treatment is also cited frequently in the literature (Hawthorne 1985) as being practical to achieve in a dry anneal and effective in reducing neutron embrittlement.

2.2 Charpy-V-Toughness

The effect of the irradiation conditions on the Cv toughness is shown in Figure 3. The curves were fitted using a tanh model to describe the experimentally determined data. The shifts in TT and changes in USE together with the tanh fitting parameter values are summarized in Table 2. Plots of the TT shifts ΔTT-41J and the changes in USE as a function of fluence for the I condition are depicted in Figures 4 and 5 respectively.

The I condition produced the well known progressive increase in the TT and decrease in the USE with increasing fluence. An estimate of the errors to be associated with the measured increases and changes of TT and USE values respectively was obtained by approximate standard deviations obtained from the plots of Figure 3.

It was evident that the forging still exhibited relatively good Cv properties even for fluences up to 5×10^{19} n/cm^2. The lateral expansion and fracture appearance criteria (not reported in detail here) also showed normal behaviour as would be expected for a nuclear quality steel with a copper content of 0.17wt%. The amount of TT shift and changes in USE were certainly within the range reported for forgings having approximately the same chemical composition and experiencing such fluence levels as used here (Pachur 1982, IAEA 1986). Compared to the predicted trends in the shift according to the USNRC Regulatory Guide 1.99, Revision 1 the ΔTT curve found in this investigation (I condition) lies well below the trend curve corresponding to 0.17wt% Cu and 0.012wt% P, also indicated in Figure 4.

3 Toughness Recovery Behaviour – Response to Annealing and Reirradiation

There are various ways of performing IAR experiments. The real question which has to be answered with IAR data is "What A treatment must be applied at fluence F in order to achieve a PLEX beyond F_{EOL} up to a fluence of F^*?" Other work in this field has tended to concentrate on the reembrittlement path (Hawthorne 1985) hence the particular choice of IAR path followed. The tendency was that a quick saturation of reembrittlement took place after irradiation and annealing (IA). However, for technological applications, only IAR with reirradiation times $t_R \sim t_I$ is of direct interest. Below we present a description of the assessed Cv toughness in the IAR condition and a phenomenological model is suggested for the way recovery and (re)embrittlement is thought to occur.

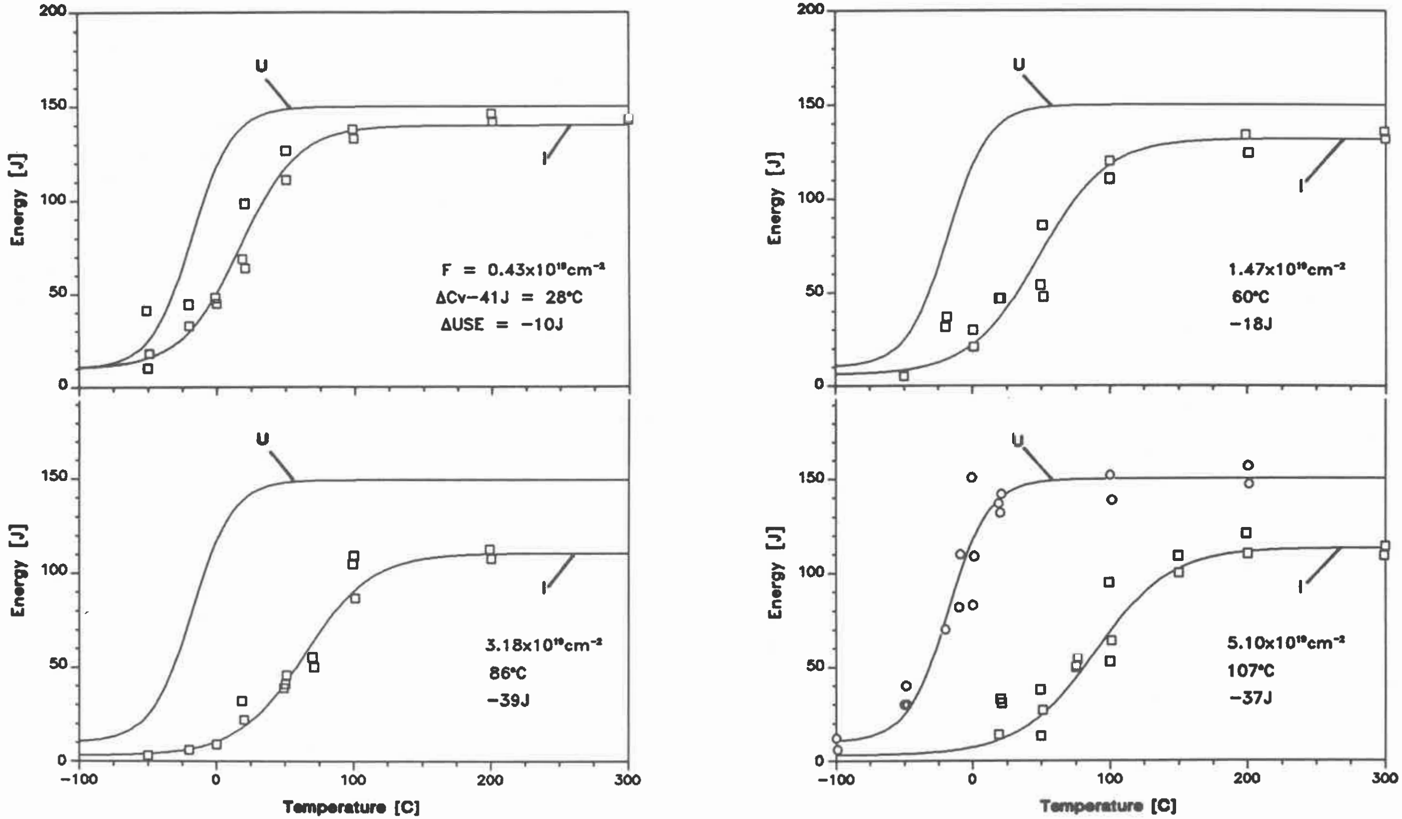

Figure 3 Charpy toughness as a function of test temperature for the unirradiated (U condition) and for different neutron fluences (I condition). The irradiation causes a progressive increase in the TT and decreases in the USE – curves were fitted using the tanh fit procedure (Table 2). After a fluence of 5.1 x 10^{19} n/cm^2, E > 1MeV at 290 C, the material still exhibited an USE of ~110 J which indicates the relatively good quality of the forging used; the lead factor was ~ 125 with respect to a typical PWR irradiation flux.

Table 2: Charpy-V properties observed for the forging with initial properties and irradiation conditions as given in Table 1.

Conditions[a]	increases[b]	change[b]	dosimetric[c]	tanh fit procedure[d]			
Cv	ΔCv41J	ΔUSE	fluence	A	B	C	T_o
	[°C]	[ΔJ]	[10^{19}/cm^2, E> 1MeV]	[J]	[J]	[°C]	[°C]
U	-	-	-	80	70	30	-18
$I_{0.5}$	28	-10	0.43	75	65	43	17
$I_{1.7}$	60	-18	1.47	69	63	50	48
$I_{3.0}$	86	-39	3.18	57	54	49	64
$I_{5.0}$	107	-37	5.10	58	55	55	89
$IAR_{0.5}$	48	10	0.47	80	80	45	35
$IAR_{1.7}$	67	-5	1.57	78	67	48	60
$IAR_{3.0}$	60	-5	2.67	75	70	50	50

a U = unirradiated, I = irradiated only, IAR = irradiated-annealed-reirradiated, the annealing schedule used was A = 450Cx168h, indices = target fluences in 10^{19} n/cm^2,E > 1MeV.

b Initial Charpy properties DBTT-41J = −37C, USE = 150J.

c Neutron fluence determination: Fe, Cu, Ni, Nb; measured lead factor = 124±10 (cf. Table 1)

d $Cv = A + B\tanh[(T - T_o)/C]$.

3.1 Experimental IAR Procedure and Cv Toughness

The IAR mode used here was based on annealing treatments after 50% of the desired end fluences had been reached (see target fluences given in Table 1). Thus $F^* = 2F$ has been used in this work. Of course, for a given total fluence F^*, treatments at any value of intermediate fluence could have been carried out and the response to annealing would be different depending on the ratio R/I in an IAR experiment.

When 50% of each of the target fluences was reached, the rigs were each time removed from the test reactor and subjected to the annealing treatment in a hot cell. A specially constructed multizone furnace was used to ensure a uniform temperature profile over the entire specimen volume of the completely loaded and instrumented rig as used in the irradiation experiments. Its instrumentation was used for temperature control purposes. The whole irradiation capsule was kept under a protective gas atmosphere. The annealing schedule used in this particular case was 168h at 450C±2C. After the heat treatment the complete irradiation capsules were returned to the reactor for the remaining irradiation up to the same target fluences as used for the I-conditions (see Table 1).

The Cv curves for the IAR condition are shown in Figure 6. The corresponding data sets were again analysed by the tanh procedure. Resulting shifts are summarized in Table 2 and Figures 4 and 5. Note that there is a tendency to saturation indicated for the IAR curve with respect to the fluence. The IAR behaviour can be characterized by a large

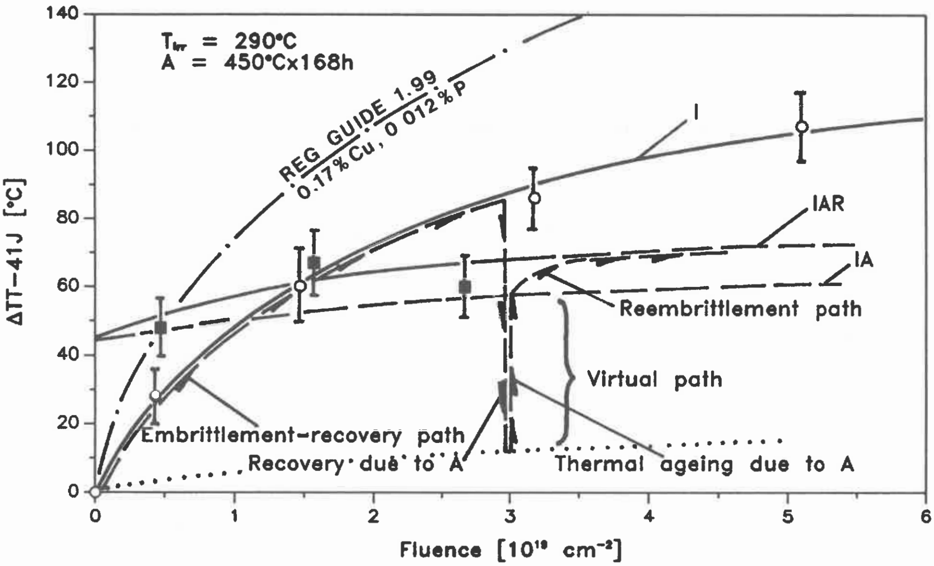

Figure 4 Transition temperature shifts as a function of neutron fluence. Both the I and IAR experimentally obtained curves are shown. The embrittlement-recovery path is also indicated together with an assumed qualitative IA behaviour which the material could exhibit. A sole thermal ageing increase of ~ 43C in the *TT* is inferred by extrapolating the IAR (IA) curves to zero fluence. The dotted line represents the virtual radiation induced *TT* increase when a thermal ageing contribution to the embrittlement would be absent. Note the existence of a vertical portion of the embrittlement-recovery path consisting of a quick recovery and a slow ageing component, both due to the A-treatment.

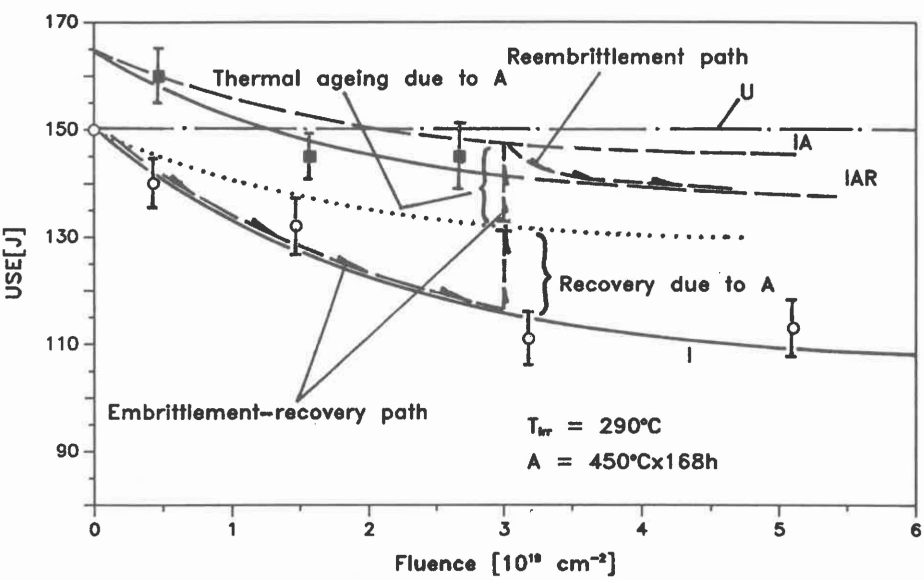

Figure 5 The *USE* as a function of neutron fluence for the I and IAR conditions. A thermal ageing effect which increases the *USE* from 150 J (U) to ~165 J (IAR) may be inferred by extrapolating to zero fluence (see also Figure 4). The embrittlement-recovery path with its contributing components (quick recovery process/slow ageing process) is schematically indicated. A probable IA behaviour is assumed. The dotted line represents the virtual irradiation induced change in *USE* (thermal ageing subtracted from IA).

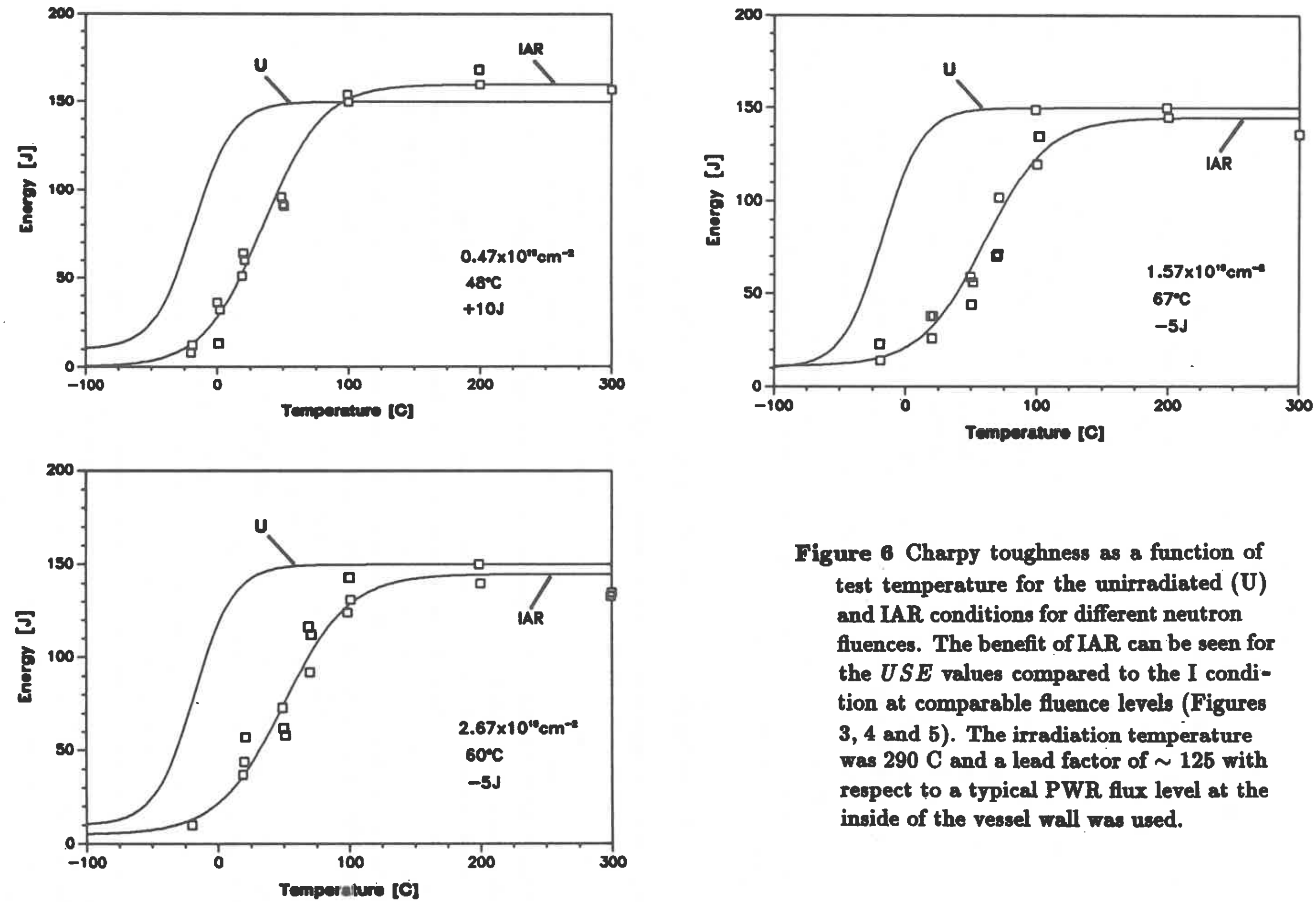

Figure 6 Charpy toughness as a function of test temperature for the unirradiated (U) and IAR conditions for different neutron fluences. The benefit of IAR can be seen for the USE values compared to the I condition at comparable fluence levels (Figures 3, 4 and 5). The irradiation temperature was 290 C and a lead factor of $\sim$ 125 with respect to a typical PWR flux level at the inside of the vessel wall was used.

benefit to the USE and, on the face of it, a somewhat smaller benefit to the TT. This general result is in accordance with other findings concerning IAR studies (Hawthorne 1985). However, if the curves of ΔTT and ΔUSE are studied closely then the following interpretation becomes evident: If the IAR - Cv curves of Figures 4 and 5 are extrapolated to zero fluence, then a contribution to ΔTT due to thermal ageing alone is indicated ($\sim$ 43°). Similarly, the IAR curve for the USE change suggests an effect of ageing, in this case beneficial, in that an increase of $\sim$ 15J is indicated over the unirradiated condition. Such an effect has been observed in other studies concerned with thermal ageing aspects of RPV steels (Hersh et al 1966). The overall behaviour can be explained by using a virtual embrittlement-recovery path model described in the following section.

For this material (composition, heat treatment schedule), this annealing schedule and this IAR procedure, it is assumed in the following that a possible benefit for PLEX is indicated by the region to the right of the fluence corresponding to $\sim$1.6x10^{19}n/cm^2, (Figure 4). The meaning of this may be explained as follows: Suppose the exposure of a PV has reached a value of 1.5x10^{19}n/cm^2. If the vessel design EOL with respect to TT shift and US toughness was at, say, 2.3x10^{19}n/cm^2, then a possible PLEX up to a fluence F^* of 3.5x10^{19} n/cm^2 would have to be planned now and realised with condition A (=450Cx168h) for the annealing treatment when the exposure of the PV will have reached the value $F = F^*/2 =$ 1.75x10^{19} n/cm^2.

3.2 Virtual Embrittlement–Recovery Path Model

Suppose an IA condition Cv-curve exists, e.g. the dashed curve in Figure 4. This takes into consideration the findings by Hawthorne (1985). Then the main model assuption is that there exists a thermal ageing effect which is dependent on the material constitution and independent of the neutron fluence (and/or of radiation embrittlement mechanisms) (Hersh et al 1966) which the material has experienced up to the point when the annealing treatment is undertaken.

At the moment, there are no results available from unirradiated aged specimens which may or may not confirm this latter assumption. It may be that the observed ageing effect is only present in irradiated material but there are indications (Mager 1978) that ageing can and does take place in unirradiated PV steels especially within the range 400-500C (Druce 1985, 1986). In the work cited, increases in the TT were more in evidence than any gains in the USE at times $\sim$ 168h. It is a fact that despite prolonged annealing at higher temperatures there still remains a potential for strain ageing, grain boundary segregation and carbide modification to take place (ageing effects) at lower temperatures for prolonged times, especially for the case of unirradiated high copper PV steels given extended annealing treatments at >425C (Mager 1978).

The indicated thermally induced increase in the TT is $\sim$ 43° (Figure 4). If this value is subtracted from the (assumed) dashed IA curve, a resultant (virtual) curve, lying entirely below the I reference curve, describes the remaining sole irradiation induced contribution to the ΔTT shift for the IA condition. The suggested (re)embrittlement paths are indicated schematically in Figures 4 and 5. Note the presence of the virtual part (quick damage recovery/slow ageing) indicated for this material. At the lower fluences a low level of radiation damage exists, the relatively large thermal ageing effect works in an opposing manner to the annealing out of the slight radiation damage. It thus causes there an overall increase in the ΔTT for the IAR condition. It was indicated by the isothermal studies that on the one hand a substantial recovery in the hardness occurred at temperatures > 440C in 1–2h, i.e. rapidly. On the other hand, thermal ageing time was 168h, i.e. for this material it was a slower process. As the fluence level increases so will the amount of radiation induced damage and, according to the model assumption, if the ageing contribution is independent of the fluence and therefore remains constant, then there will be a point where the recovery due to irradiation induced shift will be more than the increase in TT due to ageing. For the present case the fluence level for this point is $\sim 1.6\text{x}10^{19}$ n/cm^2 (see Figure 4).

For small fluences, the ageing process had caused an increase in the USE of $\sim$ 15 J above that of the unirradiated condition (Figure 5). Using similar arguments as for the case of the ΔTT, if the ageing contribution to the USE is subtracted from the (assumed) dashed curve the resultant (virtual IA) curve will describe solely the remaining radiation induced effect on the USE and it lies still above the I curve for all fluence levels. It is indicated that for the case of the USE, the ageing effect is beneficial and for the ΔTT detrimental, especially at lower fluence levels. Conversely, the net result of the effect of IAR is represented by a considerable benefit for ΔTT when the ageing effect is ignored (reembrittlement path saturating somewhat above the dotted curve in Figure 4). For the USE, benefit is immediately evident but it is somewhat less when the beneficial ageing effect is neglected (reembrittlement path saturating somewhat below the dotted curve in Figure 5).

In the case of a weldment investigated earlier (Tipping, unpublished work) there was no indication of an ageing effect in the material (0.3wt%Cu) irradiated to $5.4\text{x}10^{17}$ n/cm^2, E > 1MeV at 290C (see Figure 7). In particular, the rehardening region found in the present work (Figure 2, II) was not detected. Hence the virtual part of the embrittlement-recovery path described in the present model would be absent. This serves to underline the importance of the material constitution in IAR studies.

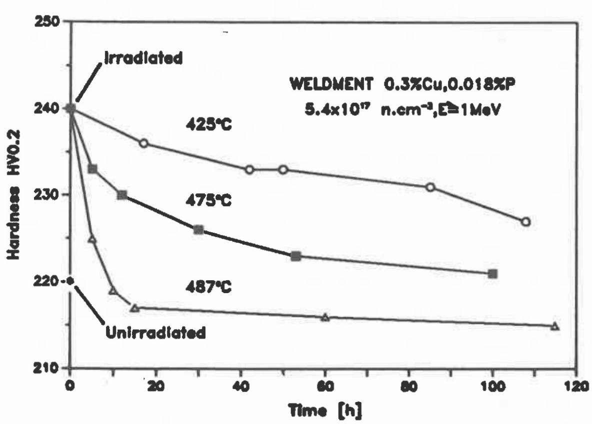

Figure 7 Isothermal behaviour of a weldment (Tipping, unpublished work). The radiation induced hardness increase was removed progressively with increasing temperature. There was no indication that any rehardening took place such as was measured for the forging. However, it should be noted that the time scales between this and Figure 2 are such that regions I and II correspond to approximately the first 20h. Furthermore, 50% recovery was evident after $\sim$ 5h at 487 C which indicated a somewhat slower annealing rate compared to the forging.

4 Summary and Conclusions

During an annealing there are two main mechanisms at work namely:

i) radiation induced embrittlement relief and

ii) thermal instability effects such as ageing and rehardening.

These phenomena will be highly material dependent (composition, heat treatment schedule) and will also be dependent to some extent on the annealing conditions (temperature, time) used. The mechanisms i) and ii) are active in opposed directions as far as the recovery in ΔTT is concerned but mutually enhancing for the recovery in USE.

According to the phenomenological model description of the embrittlement-recovery path in an IAR procedure, there will be a virtual path contribution due to the above mentioned mechanisms i) and ii). In case of (partial) absence of the ageing mechanism ii) it is to be expected that the properties ΔTT and ΔUSE will change their role as far as the extent of recovery is concerned.

IAR procedures can be beneficial for PLEX according to the preliminary results reported here, provided an irradiation dose rate effect does not influence the recovery to be expected. It should be noted that these studies have been carried out using flux levels ~125 times that encountered at the inside vessel wall of a commercial pressurized water reactor.

IAR experiments must be carried out in order to determine the plant specific optimal annealing performance for the PV. As long as IAR can be demonstrated as being of long term benefit for PLEX, the reembrittlement path is of less importance as far as the technological application of a PV annealing is concerned.

For the present material and irradiation conditions and the chosen IAR mode, a PLEX beyond an EOL fluence typical of PWR's can be obtained if annealing is carried out at a fluence which is ~50% of the anticipated target fluence for PLEX.

Further studies are in progress to elucidate the material's behaviour in the unirradiated but annealed condition (450Cx168h), i.e. thermally aged. A specimen set at a nominal fluence of 5×10^{19} n/cm^2 has already been subjected to the above mentioned annealing procedure.Test results for this fluence will be available during 1987.

An experiment will be carried out using the same IAR mode as described in the present work. However, another annealing temperature (460C) will be used for a short time (1-2h) which should correspond to a rapid recovery of the radiation induced damage but should largely avoid any thermal ageing contribution (region I, Figure 2). Accordingly, a larger benefit to the ΔTT recovery and less benefit to the USE is expected.

An effort will be made to identify any ageing mechanisms at work (e.g. segregation of phosphorus to grain boundaries, etc.) during a 450 C annealing treatment. To this end, physical examination methods (SANS, DENS, e^+-annhilation, etc. see Solt et al, this conference section G) will be used.

Other mechanical tests such as tensile and fracture mechanical testing will be undertaken to identify any further effects of IAR.

Acknowledgement

The valuable assistance given by Mr. A. Christen concerned with Charpy testing in the hot cell is acknowledged.

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