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EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

STEELS WITH REDUCED SENSITIVITY TO NEUTRON-EMBRITTLEMENT : A SAFETY FACTOR

by

J. SEBILLE

1967

EURATOM/US Agreement for Cooperation

EURAEC Report No. 1883
Directorate-General for Research and Training, Brussels-Belgium

Paper presented at the Panel of Experts on Recurring Inspection
of Nuclear Reactor Pressure Vessels
Pilsen - Czechoslovakia, October 3-7, 1966

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Printed by Guyot, s.a.
Brussels, September 1967

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SUMMARY

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KEYWORDS

NEUTRON BEAMS
RADIATION EFFECTS
STEELS
BRITTLENESS
FERRITE
GRAIN SIZE
METALLOGRAPHY
STAINLESS STEELS
HEAT TREATMENTS
NITROGEN
DEFECTS
REACTORS
PRESSURE VESSELS
MECHANICAL STRUCTURES

Dislocations
interstitials
transition temperature

CONTENTS

INTRODUCTION

Reactor vessels have increased in size gradually over the last ten years or so to keep pace with the increase in power developed by reactors, as can be seen from Fig. 1 in the case of pressurized-water reactors (PWR). At the same time a similar trend is to be observed with regard to their thickness (Fig. 2). However, this trend cannot continue indefinitely for various reasons of a technical, technological and practical nature, such as the greater difficulty with regard to rolling, bending, welding and non-destructive testing, etc., the increase in the thermal gradients through the thickness and in the associated stresses, the increased danger of brittle fracture, the impossibility of transporting or even handling reactor vessels above a certain weight or dimension, etc. For these reasons other solutions have had to be considered, such as multilayer vessels, vessels of high-strength quenched and tempered steel, etc.

However, the choice of the most suitable steels is primarily governed by their resistance to neutron bombardment. While numerous experimental results have provided us with an empirical knowledge of steels under irradiation, there has so far been no overall systematic and analytical study which has led to the scientific design of steels offering better resistance to this type of embrittlement.

This is of primary importance, however, for the useful life of the vessels could be extended for equal reactor power, or the specific output of installation could be stepped up for an equal useful life of the vessel. Finally, periodic inspection of reactor vessels would be considerably simplified once the drawbacks and uncertainties of neutron embrittlement were eliminated. It would thus appear useful to attempt to pinpoint the metallurgical parameters of neutron embrittlement in order to map out the best way of developing new steels which are less sensitive to it.

The main aim is still to reduce the shift in the transition temperature due to irradiation, but it should not be forgotten that

Manuscript received on October 5, 1966.

certain undesirable phenomena can take place at the vessel's operating temperature (e.g., thermal embrittlement) and that the steel should not be affected by them. Finally, in view of the fact that "defect-annealing" may have to be carried out on the vessel after a certain operating time, the steel should lend itself easily to the recovery of its properties and should not undergo thermal embrittlement during this annealing process.

In addition to these requirements, which are related directly to the creation of irradiation defects, it goes without saying that the steel should be as resistant as possible to all types of embrittlement throughout all the stages in its manufacture and use.

I. REDUCTION OF Δt*

For a long time now metallurgical parameters have been regarded as having only a minor, or even negligible effect on the irradiation embrittlement; the Δt values corresponding to a given integrated flux for a large number of steels all fall within a scatter range whose width depends, it is felt, almost entirely on the uncertain factors involved in dosimetry and on the diversity of the test specimens used. This was the case with the microstructure, the grain size and mechanical ageing.

(a) Ferritic Grain Size

Using the relation of Petch (Ref. 1), Churchman et al. have nonetheless shown theoretically (Refs. 2 and 3) that fine-grain steels can withstand a higher integrated neutron dose than coarse-grain steels without causing brittle fracture. This has been corroborated experimentally by Mogford (Ref. 4) for irradiation temperatures of 100 and 150°C. The results obtained by other workers (Ref. 5) are given in Table 1 (integrated dose $7 \cdot 10^{17} n/cm^2$).

Slightly different results were obtained by Cibois, and by Berggren and Wilson (Ref. 6).

* Δt denotes the shift, caused by irradiation, of the impact-strength transition temperature of the steel, this corresponding, for example, to a certain rupture-energy level in a Charpy V-notch test.

Table 1

Steel	No. of grains per mm^2	Δt (°C) corresponding to 35 ft-lb
Steel Al-killed mild steel	523 6,890	50 5
Si-killed mild steel	76 842	65 45
Ni-Mo-V steel	5,060 36,500	40 10

More recently, Myers, Grounes and Hannerz (Ref. 7) confirmed the considerable superiority of fine-grain steel on a carbon steel which was treated so as to obtain austenitic grain sizes of 9 and 6.5 ASTM. Using miniature samples, they found a Δt value for fine-grain steel which was more than twice that for coarse-grain steel, the shift in the transition temperature being measured at the energy-level corresponding to the NDT of the steel.

(b) Influence of Microstructure

With regard to the microstructure, a parameter which had been ignored up to then, Cibois et al. (Ref. 8) showed that, after irradiation at 180-200°C up to a fast integrated dose of 5 to $6 \cdot 10^{18}$ n/cm^2, one and the same MnMo steel treated so as to have a fairly large range of microstructures revealed Δt values varying from 52 to 193°C, the minimum corresponding to a quenched and tempered structure and the maximum to an overheated structure. This confirmed, in particular, the superiority of quenched and tempered structures as pointed out by Trudeau (Ref. 9) for a normalized, austempered or quenched and tempered HY-65 steel. After irradiation up to a fast integrated dose of $3.4 \cdot 10^{19}$ n/cm^2, a Δt of 243°F (135°C) was observed for the quenched and tempered state and 355°F (199°C) for the normalized condition, the "austempered" state being between the two previous ones. In addition, the very competent study carried out by Carpenter, Knopf and Byron (Ref. 10) revealed the existence of considerable differences in irradiation behaviour between various heats of the same steel, particularly with regard to the impact strength, to such an extent that it was possible to draw a distinction, as shown in Fig. 3, between "sensitive" and "insensitive" heats. The first showed a Δt of 425°F (235°C) for a fast integrated dose of about $5 \cdot 10^{19}$ n/cm^2, while the value for the latter is only 170°F (95°C). The main factor accounting for the difference is the heat treatment applied, consisting of normalizing or quenching, followed in some cases by tempering and even cyclic tempering. Apart from slight differences in the chemical composition which are not thought to play a major role, it would appear that the microstructure is the prime factor accounting for the differences observed, the "sensitive" heats exhibiting coarse-grain bainite-ferrite structures which are sometimes distributed in bands, while the "insensitive" heats have a fine-grain and a tempered ferrite-low-bainite microstructure. Moreover, generally speaking, it is plates which originally had the lowest transition temperatures which are regarded as "insensitive". However, there are enough exceptions to this rule to justify it only being regarded as the manifestation of a general trend on which more detailed studies are required.

Finally, recent data obtained by the Naval Research Laboratory (Ref. 11) as a result of a single irradiation experiment carried out on two heats of A-302-B steel showed that the one heat had a Δt of 225°F (125°C) while the other showed a value of 320°F (175°C), or 40% more.

Under the Yankee vessel inspection programme, Steele et al. (Ref. 12) carried out simultaneous irradiations on Charpy V-notch samples of five high gauge plates of ASTM A-302-B steel at 550°F (290°C) at an integrated dose of $3 \cdot 10^{19}$ n/cm^2 ($>$1 MeV), one representative of the steel used in the Yankee vessel, one taken from the stock of A-302-B reference steel and three from other plates of the same grade.

Table 2

Steel A-302-B	Charpy V-notch transition temp. at 30 ft-lb (5.2 kgm/cm^2) before irradiation	after irradiation	Δt(°F)
Reference	30	200	170
Yankee			
Sample 1	55	260	205
Sample 2	105	260	155
Heat A	25	160	135
Heat B	-15	125	140
Heat C	30	150	120

The following observations thus emerge:

- A considerable difference in the transition temperatures of different steels both in the non-irradiated (maximum scatter 120°F) and the irradiated state (maximum scatter 135°F);
- Considerable differences in the transition temperatures for two adjacent samples taken from the same plate 7 $^7/_8$" thick;
- A maximum scatter of 85°F between the transition temperature shift values between the various steels after irradiation.

After metallographic inspection, it was observed that while the normal microstructure of this steel is a fairly fine tempered bainite, certain plates have a coarse upper bainite structure (Yankee sample 2) while heats A, B and C are largely spheroidized.

The structural factor certainly seems to be the main cause of the differences observed in this case.

In a recent series of experiments the same workers also demonstrated that the irradiation behaviour of a steel can be substantially changed by modifying its microstructure, all the other metallurgical parameters remaining constant. By way of an illustration, it was possible to invert the order of merit of two steels, one of them (HY-80) being regarded as relatively insensitive to irradiation, and the other (A-350-LF3) being known to be very sensitive, by means of simple heat treatment, so much so that the microstructure can be considered as being the main factor governing the extent of the irradiation damage. In the present case the best structure is once again tempered martensite containing a few traces of ferrite, the austenitic transformation products formed at high temperatures being less favourable.

(c) Influence of the Composition

Very few studies are available on the effect which the chemical composition of steels can have on their irradiation behaviour. This can probably be accounted for by the fact that, since the representative points of the damage as expressed in $\Delta t°$ (increase in the transition temperature) as a function of the logarithm of the integrated dose are all in a narrow, straight scatter range, it was readily concluded that, apart from experimental errors, the main factor involved, if not the only one, was the integrated neutron dose. This would explain the absence of any large-scale systematic studies on these specific effects of the various elements and would also account for the need to calculate them on the basis of comparisons of the irradiation results of various steels, for which the composition is given in full. This work has recently been carried out by Francke and Garzarolli (Ref. 13), to whose publication reference should be made.

We will limit ourselves here to enumerating recent results, which, if corroborated in practice, show that either the composition or the joint action of the composition and the microstructure could be taken as a basis for designing steels which are virtually insensitive to irradiation, the main alloying element used being nickel.

Influence of Nickel

The use of nickel is an attractive proposition because it enables the steel to be given a very low transition temperature in the non-irradiated state. Provided that it does not, when irradiated, cause a greater shift in the transition temperature than that observed in steels containing no nickel, this element can be used in reactor vessel steels. Unfortunately, there is still some doubt as to its actual effect, some workers being of the opinion that it has a favourable influence, while others are of the opposite view. Porter (Ref. 14) has already pointed to the study carried out by Trudeau on alloyed ferrites, where nickel offered obvious advantages in the non-irradiated state, which, however, were not maintained after irradiation.

Steele and Hawthorne (Ref. 15), extending studies of nickel additions to cover steels, have shown that the irradiation damage increases with the nickel content up to about 6 wt.%, but that beyond this value it is lower than for steels containing no nickel.

Comparison of other results obtained by Trudeau (Ref. 16) and Steele and Hawthorne (Ref. 17) shows that in one case a Δt value of 150°C is obtained with a dose of $1 \cdot 10^{19}$ n/cm^2 ($>$ 1 MeV) and in the second a Δ t of 85°C for a dose of $7 \cdot 10^{18}$ n/cm^2 ($>$ 1 MeV). These values are no different from those found for steels containing no nickel. It should, however, be pointed out that the irradiation temperatures (50-70°C and 115°C respectively) are without doubt too low to cause the embrittlement frequently encountered in other tests.

In another study (Ref. 18) a steel containing 9% nickel yielded Δt values which were much higher than those for mild steels at irradiation temperatures of 155 and 330°C. Furthermore, in the second case, the shift amounted to 205°C for a dose of $0.6 \cdot 10^{19}$ n/cm^2 as against 30-60°C for steels containing no nickel. This nickel-bearing steel would, therefore, not only be very sensitive to irradiation but also to thermal embrittlement at high irradiation temperatures.

In a recent study (Ref. 19) various steels were compared, some of them being experimental materials. Some of the results obtained are given in the following table.

Table 3

Steel and initial t_{tr}		Δt (°F) Irradiation at 240°F $1.1 \cdot 10^{19}$	Δt (°F) Irradiation at 550°F $3.8 \cdot 10^{19}$
A-212-B	5°F	245	185
A-302-B	30	-	160
HY-80 (3% Ni)	- 205	170	-
3% Ni-Cr-Mo	- 105	205	-
3.5 Ni-Cr-Mo	- 130	210	90
7.5 Ni-Cr-Mo	- 215	130	15
A 353 (9% Ni)	- 370	245	175

It can be seen that at 550°F (290°C), i.e., a temperature which is representative of the vessel's operating conditions, the two Ni-Cr-Mo steels show a very low Δt value compared with the others. That for the 7.5 Ni-Cr-Mo steel is negligible and this steel can be classified as "insensitive" to irradiation, at least for high irradiation temperatures.

It will be noted that A-353 steel, which contains 9% Ni but no Cr or Mo, is not nearly as good, which proves that the good behaviour of the two previous steels is not entirely due to the nickel content. Until a complete analysis has been performed on these steels, it is impossible to say whether this can be accounted for by the method of fabrication used.

To sum up, the effect of nickel, about which relatively little is known, seems to be favourable at certain contents and when used under irradiation conditions which do not cause thermal embrittlement.

(d) Influence of Fabrication Method Used

Examination of the numerous results obtained so far on the shift of the transition temperature of rolled, forged or welded steels reveals that they are all located in a fairly narrow scatter range (see Fig. 4) and that it would be deceptive to try and devise a systematic method for classifying the different manufacturing techniques.

However, on various occasions it has been thought that some advantages were to be gained from a particular method of deoxidation or a particular technique for degassing the steel. Trudeau (Ref. 20), for example, working on quenched and tempered Ni-Cr steels, compared commercial products with steels which had been treated in a vacuum and then cast in a vacuum. Although the gas contents were unfortunately not determined, the elements which differed markedly from one product to another were the following:

wt.%	S	P	Mn
High-purity heat	0.002	0.002	0
Commercial heat	0.014	0.014	0.36

It was found that for an integrated dose of $5 \cdot 10^{19}$ n/cm^2 ($>$ 1 MeV) the Δt value for high-purity steel was only about 75% of that of commercial steel.

Moreover, the possibility cannot be ruled out that there is a systematic difference in the behaviour of heats made normally (in air) but deoxidized with either silicon or aluminium. Although not enough results are available to prove this, the tensile behaviour of the two types of steel indicates a correlation between the hardening of aluminium-killed steels and the irradiation temperature which is not observed in silicon-killed steels.

The method used for deoxidizing and killing the steels probably has its effect via the nitrogen, either in the free or combined state. It has, in fact, been observed that, as in the case of aluminium-killed steels, the Δt value for steels containing vanadium was higher for an irradiation temperature of 130°C than for 80°C (66 and 35°C respectively for vanadium-containing steel), while this peculiarity is never found in silicon-killed steels.

It would also appear (Refs. 21 and 22) that a silicon content of 0.5 to 0.75% (and perhaps more) stabilizes the Δt at a high and almost constant value for all irradiation temperatures up to 400°C, except around 250°C, where Δ t is lower.

It is worthwhile pointing out that the trend towards the use of quenched and tempered steels may enhance the value of silicon, since, in order to allow for a particular type of tempering embrittlement (at low temperature, i.e., that which occurs between 200 and 375°C), enough silicon (0.5 to 1%) has been added to some of these steels in order to shift the embrittlement towards higher temperatures, thus enabling higher tempering temperatures to be used (which, in some cases, gives them the additional advantage of secondary hardening). The first such steel to be made was HY-TUF (Ref. 23). It will thus be best to use only low silicon contents, at all events less than 0.5%, if one wishes to remain within the range of medium-strength low-alloy steels.

The Naval Research Laboratory (Ref. 24) recently undertook a systematic study of different metallurgical parameters, including, in particular, the fabrication method. Five heats of A-302-B steel were compared before and after irradiation (irradiation around 100°C at a dose of $3 \cdot 10^{19}$ n/cm^2).

Table 4

Material	Heat treatment	Transition temperature before	after	Δt °F
A. Commercial heat	Austenitization and tempering	25	335	310
	idem + stress-relief annealing*	30	345	315
B. Induction furnace with residuals	Aust. and tempering	65	350	285
	idem + stress-relief annealing	60	340	280
C. Induction without residuals	Aust. and tempering	-35	235	270
	idem + stress-relief annealing	5	245	240
D. In vacuum without residuals	Aust. and tempering	55	240	185
	idem + stress-relief annealing	145	340	195
E. In vacuum without residuals with 1.35% Ni	Aust. and tempering	70	340	270
	idem + stress-relief annealing	145	395	250

* The stress-relief anneals were carried out for 30 hours at 610°C. The residuals were P,S, Ni, Cr, Cu, Sn, etc.

The first conclusions to be drawn from this study, which is still in progress, are:

- The residual elements do not appear to play any part in the heats made in air (B and C) in view of the similar Δt values, especially in the non-stress-relieved state;

- Heat D shows a considerably lower Δt value, which would suggest that the gas contents have a marked influence and which would, in addition, indicate the appeal of steels which are made and cast in vacuo, although this latter view has yet to be confirmed.

(e) Influence of Previous Tempering Embrittlement

The question asked was whether treatment in the form of temper embrittlement before irradiation could change the steel's sensitivity to irradiation and, in particular, the variation in the transition temperature measured during the impact test. This question is of obvious practical interest in view of the fact that large welded assemblies such as reactor vessels must undergo stress-relief treatment after partial or total welding. In such cases it frequently happens that the treatment, whether one-time or repeated, can be of a total duration of several tens of hours or even 100 hours or so at temperatures of the order of 550 to 650°C. It has often been observed that such treatments can cause a major reduction in the impact strength before the vessel is put into service.

Steele, Hawthorne, Serpan, Watson and Klier (Ref. 25) used an Ni-Cr-Mo steel of the following composition:

C 0.13 - Mn 0.34 - Si 0.26 - Ni 4 - Cr 1.71 - Mo 0.49 - Al (soluble) 0.002

N 0.008 - S 0.022 - P 0.012

treated in two ways:

A) austenitized at 815°C, quenched in water, tempered for one hour at 650°C and quenched in brine;

B) austenitized at 815°C, quenched in water, tempered for one hour at 650°C, oven-cooled down to 480°C at the rate of 35°/hr, held at temperature for one hour, oven-cooled down to 315°C at the rate of 35°/hr and cooled in air.

Treatment B was effective since it imparted to the steel an embrittlement which manifested itself in an increase in the transition temperature (defined by the Charpy V energy level 5 kgm/cm^2) of 70°C in the non-irradiated state. After irradiation at the fast dose of $1.8 \cdot 10^{19}$ n/cm^2 at a temperature of 115°C, the same difference is observed between the transition temperatures of the two steels, as well as the same maximum energy level.

Provided that these observations are confirmed in the case of other steels, the conclusion to be drawn is that tempering embrittlement prior to irradiation in no way effects the neutron embrittlement, its only effect being to raise the transition temperature of the steel before irradiation.

(f) Influence of Strain Ageing

The influence of previously induced plastic deformation has a also formed the subject of studies, since it can happen that the steel is subjected to a plastic deformation of several per cent during fabrication or in the course of the hydrostatic test.

Both Nichols and Harries (Ref. 26) and Cibois et al. (Ref. 8) found that deformations of this order did not alter the Δ t value due to irradiation. Proceeding along the same lines, Grounes and Myers (Ref. 27) demonstrated that strain-ageing has the same effect on the transition temperature of steel before and after irradiation, its effect merely adding to that of the irradiation.

*

* *

To sum up, the experimental results quoted suggest that the Δt value could be reduced if the following points are borne in mind:

1. The practice of using fine-grain killed steels can be regarded as well-founded and advisable;

2. An attempt must be made to cut down or eliminate segregations in high-gauge plates and variations in properties between different heats of the same steel;

3. Studies must be continued on the influence of nickel, which appears to be an interesting element when used in moderate quantities (3-7%) in conjunction with chromium and molybdenum;

in particular, systematic studies should be performed on the microstructure and the residual austenite content of these steels;

4. The trend aimed at should be towards high-purity steels having very low contents of gases, and possibly residuals, which latter have a damaging effect, especially in the case of quenched and tempered steels of medium to high strength (Ref. 28);

5. Steels whose microstructure contains austenitic decomposition products formed at high temperature show less satisfactory neutron irradiation behaviour than those having a quenched and tempered structure; this latter should be studied at the same time in order to provide the best combination of impact strength in the non-irradiated state and transition temperature shift due to irradiation;

6. Finally, an attempt should be made to find the optimum combination of quenched and tempered structure and high purity (especially in gases).

II. REACTION TO "DEFECT-ANNEALING" AND POSSIBILITY OF IMPACT STRENGTH RECOVERY

1. The Effect of "Defect-Annealing"

Examination of the transition temperature shifts measured hitherto shows that they can amount to nearly 300°C for integrated doses such as those likely to be encountered in power reactor vessels after 20 or 30 years in operation. For example, an A-350-LF3 steel (containing 3.25% Ni and 0.04% V) gave a Δt value of 275°C after irradiation at 150°C up to a fast integrated dose of $4 \cdot 10^{19}$ n/cm^2 ($>$ 1 MeV). In such a case the desirability of recovery, albeit partial, of the impact strength properties is obvious.

In actual practice, one method which could be used would be to carry out the annealing with the aid of the vessel cooling water when the treatment temperature remains below 340-350°C, above which helium or argon could be used.

It should be noted in passing that, in the theoretical field, the studies on the kinetics of recovery due to post-irradiation annealing are also interesting owing to the fact that they may help to provide a better understanding of the mechanism of irradiation damage.

The first recovery studies, which concerned the yield strength, showed that this process commences around 250°C, its efficiency increasing with the annealing temperature up to about 450°C, by which time the recovery is generally complete.

Nichols and Harries (Ref. 29) have demonstrated that, in a diagram giving the logarithms of the isothermal annealing time in the abscissa and the elastic limit in the ordinate, the phenomenon is represented by parallel straight lines, indicating that its activation energy is constant for all annealing temperatures. It is thus fairly easy to find "equivalent" conditions for recovery treatments. Moreover, if it is assumed that the phenomenon varies exponentially with time, it can be used to deduce the value of this activation energy and at the same time it is possible, with the aid of one single straight line, to represent all the temperature-time recovery combinations which yield a given percentage of recovery (the inverse of the absolute temperature in the abscissa and the logarithms of the annealing time in the ordinate).

Still using the yield strength measurements, the same authors also observed:

- that if the irradiation temperature is raised above a certain value (150°C in their tests) for the same integrated dose, longer annealing times are required to obtain the same percentage of recovery (which would tend to confirm the theory that the nature of irradiation defects varies with the temperature at which they were formed);

- that the same applies if the integrated dose is increased for a given irradiation temperature.

In another study, carried out by the Bettis Laboratory (Ref.10) the percentage of recovery was evaluated on the basis of the impact transition temperatures and for annealing treatments of 9, 48 and 72 hours at three temperatures, namely, 345, 385 and 425°C.

The following observations emerged:

- the higher annealing temperatures give the better recovery values;

- the longer times at these temperatures give lower recovery values;

- "irradiation-sensitive" heats respond less well to treatment than "insensitive" heats, often in the ratio of 1 : 2, despite the fact that the absolute gain with respect to the Δt value (expressed in degrees C) is greater for the former;

- the annealing treatment can have very different effects on heats which showed the same Δt value after irradiation.

The majority of these results are in good agreement with those obtained by the Naval Research Laboratory, which has for several years now been carrying out the most thorough studies so far performed on this problem (Refs. 30 and 31).

Table 5 shows the influence of the annealing temperature and time on the impact strength recovery and the reduction in the Δt caused by irradiation of a forged steel.

Table 5

Impact strength recovery - A-350-LF1 steel after irradiation at 221°C at 3.1. 10^{19} n/cm^2 (> 1 MeV)(Δ NDT obtained by irradiation = 246°C)

"annealing" conditions		Obtained by recovery		Residual Δ NDT °C
Temperature °C	Time h	NDT (°C) after recovery	% improvement	
260	18	31	13	215
260	168	73	30	173
316	1	92	38	154
316	18	162	65	84
316	168	193	79	53
399	1	224	91	22
399	18	238	97	8

The data given in Table 6 below confirm that the higher the temperature at which irradiation is carried out, the lower will be the percentage of recovery from irradiation damage for a given anneal, even if this recovery is greater in absolute terms.

On the other hand, if it is decided to anneal the same steel at a temperature which is invariably 50 to 100°C higher than the irradiation temperature (and for a constant integrated dose), the most complete recovery is generally found for the highest irradiation temperature.

Table 6

State		Transition temperature °C	Δt (°C) with respect to State A	% recovery
A	Non-irradiated	-13	-	-
B	Irradiated at 135°C	145	158	-
BR	Annealed for 36 hr at 400°C	8	21	86%
C	Irradiated at 266°C	100	113	-
CR	Annealed for 36 hr at 400°C	35	48	57%

"Defect-annealing" treatment thus offers a convenient way of restoring the properties of an irradiated vessel in such a way that its useful life can be prolonged considerably at the cost of one or more annealing operations. However, the treatment of each vessel will have to be decided on its merits, not only because, as was seen above, the degree of recovery depends on the type of steel, the irradiation temperature, the dose received and the annealing conditions, but also owing to the occurrence, in certain steels, of a re-embrittlement phenomenon, probably of thermal origin, which takes place as a result of long-time annealing.

2. Thermal Embrittlement

It has been seen that damage caused to a metal by irradiation can be more or less entirely corrected by subsequent heating, when it is known as "defect-annealing". In the same way, when irradiation takes place at increasing temperatures, the damage tends to decrease gradually, finally reaching a zero value for an irradiation temperature which is high enough to cause "self-annealing" of the defects or self-recovery of the properties of the metal. Table 7 shows the gradual decrease in Δt for rising irradiation temperature in two different steels at the same integrated dose.

Table 7

Increase in NDT for Steels Irradiated at Different Temperatures

Steel	Irradiation temperature	Neutron dose (n/cm^2: >1 MeV)	Δ NDT °C
A-212-B plate 4" thick (101.6 mm)	127	$6.6 \cdot 10^{18}$	118
	204	$6.6 \cdot 10^{18}$	101
	232	$6.6 \cdot 10^{18}$	112
	288	$6.6 \cdot 10^{18}$	56
A-302-B plate 6" thick (152.4 mm)	127	$5.0 \cdot 10^{18}$	95
	204	$5.0 \cdot 10^{18}$	73
	232	$5.0 \cdot 10^{18}$	78
	288	$5.0 \cdot 10^{18}$	36

However, there seem to be certain exceptions to this tendency, which is observed in numerous steels, as is borne out by Table 8, where it can be seen that the highest irradiation temperature results in maximum damage for the second steel. Now that this finding has been confirmed, it would appear that unanticipated embrittlement, probably of thermal origin, can occur in certain steels and be falsely attributed to irradiation. This fact is of great practical importance with regard to the development of better steels for reactor vessels, which should not be marked by phenomena of this kind.

It was also found that in some cases "defect-annealing" led to similar embrittlement for a certain time at the "annealing" temperature.

Table 8

Increase in NDT for Two Steels Irradiated at High Temperatures at a Neutron Dose of $3.1 \cdot 10^{19}$ n/cm^2 ($>$ 1 MeV)

Steel	Irradiation temperature °C	Δ NDT °C
A-302-B plate 6" thick (152.4 mm)	282 338 393	92 62 36
HY-80 (Ni-Cr-Mo) plate 3" thick (76.2 mm)	282 338 393	81 62 /126/

Table 9 provides an illustration of this phenomenon in a sheet of 100 mm A-212-B steel irradiated at 150°C up to an integrated dose of $1.3 \cdot 10^{19}$ n/cm^2 (1 MeV) and annealed for varying periods at 320°C. Re-embrittlement is found to occur after 28-168 hours' annealing.

Table 9

State	Charpy-V notch transition temperature 30 ft-lb (°C)	Δt °C
Non-irradiated	-10	-
Irradiated at 150°C	130	140
Annealed at 320°C:		
- 5 hours	80	90
- 20 hours	8	18
- 168 hours	52	62

It was also reported that a quenched and tempered HY-80 steel 200 mm thick underwent major re-embrittlement if it was annealed for 168 hours at 450°C after irradiation at 375°C.

The transition temperature for the 30 ft-lb value is in this case raised from -80°F in the irradiated state to -10°F in the "annealed state".

The effect of protracted heat treatment on different irradiated steels was studied for "annealing" temperatures of 345, 400 and 480°C (Ref. 32) on the basis of the variation in transition temperature at 30 ft-lb. The treatment lasted 2400 hours.

Table 10

Steel	345°C	400°C	480°C
A-212-B	No effect	30°F increase	No effect
A-302-B	No effect	No effect	No effect
HY-80	20°F increase	85°F increase	No effect

A fairly curious observation made in the case of HY-80 steel is that comparison of various tests carried out both at Hanford and at the Naval Research Laboratory sometimes gives the impression that irradiation promotes "annealing" embrittlement and sometimes indicates that it reduces it.

However that may be, this type of embrittlement manifests itself at certain irradiation temperatures and under certain "defect-annealing" conditions. What are the reasons for it?

Firstly, it will be noted that, as indicated by the data available, this embrittlement mainly affects quenched and tempered steels of high yield strength. It will also be seen that the high-strength steels now being developed are in ever-increasing numbers either quenched and tempered or spray-quenched and tempered. Various studies have shown that in the non-irradiated state such steels may be sensitive to the effect of prolonged residence at sub-critical temperatures, even if these are low.

In particular, while it is known (Ref. 33) that spray-quenched and tempered steels are generally better than normalized and tempered steels, even after cold-working, ageing or prolonged stress-relief annealing, little is known about the effect on them of protracted heat treatment at temperatures near those at which reactor vessels operate or those at which they could be "annealed" after irradiation.

In a major study devoted to this problem, Pense, Gross and Stout (Ref. 34) subjected nine steels - two carbon steels (A-285 and A-212) and seven high-strength steels (48S5, A-302, A-203, HY-65, A-387, HY-80 and T1) to treatments of 2000-8000 and 16,000 hours at 260 and 370°C. For the temperature of 260°C, the embrittlement obtained is, generally speaking, more or less negligible. At 370°C, on the other hand, it may lead to a rise of 55°C in the transition temperature and mainly occurs in steels containing nickel, the most sensitive of which are HY-65, HY-80, A-203 and T1). This embrittlement, as well as its elimination, varies with the previous thermo-mechanical history of the steels.

In another study (Ref. 35) increases of about 55°C were also observed after residence times of 15,000 hours (two years) at temperatures of about 300°C (operating temperature of certain light-water vessels) or after times of half that length for temperatures in excess of 400°C (A-203 steel).

Although less pronounced, embrittlement occurs much more quickly in other steels, e.g., after about one hour at 300°C for T1 and after two or three hours at the same temperature in HY-65.

It was not a complete surprise to find that it is the steels containing the greatest percentages of alloying elements, and hence possessing the highest hardenability, which are most subject to this embrittlement, which may be, at relatively low temperatures (250-400°C), a form of temper embrittlement. There is still a great deal of conjecture with regard to its causes, but the general consensus is to attribute it to the presence of impurities such as phosphorus, arsenic, antimony, tin and nitrogen (Ref. 36).

Although we have no figures for the temperatures around 300°C, it will be recalled that Steven and Balajiva found that the addition of 0.05% antimony causes an increase of more than 300°C in the transition temperature of low-alloy Ni-Cr steel after a good hundred hours at 450°C.

Among the elements which have a marked influence, although less pronounced than that of antimony, for the contents generally encountered in commercial steels, special mention should be made of phosphorus and manganese.

There are two known methods for developing steels which are not marked by this type of brittleness:

- the use of high-purity steels (Ref. 37) such as those cast in vacuo and specially purified;

- the addition of molybdenum, which reduces this brittleness without eliminating it.

However, this last element has the drawback of causing irreversible temper embrittlement (which may occur during the stress-relief annealing of reactor vessels) for shorter times and lower temperatures (Ref. 38). If we turn to the first method, the use of high-purity steel, this would mean that it would have to be produced on an industrial scale as economically as possible, while its welding would require filler products of the same quality and the use of welding techniques which preclude contamination during metal transfer through the electric arc. A major problem has therefore still to be solved.

III. THEORETICAL MEANS OF REDUCING IRRADIATION DAMAGE

Basic Principles

Any modification of the metallurgical parameters aimed at reducing the shift in the transition temperature must be warranted *a priori* by a valid mechanism of interaction with the defects due to irradiation.

It will be recalled that these can appear in the form of vacancy-interstitial pairs and by point defects, either isolated or in clusters. The behaviour of these clusters is complex and depends on the structure of the metal, the different interatomic forces present, the temperature, etc. They can bring about major changes in the mechanical behaviour of the metal by impeding the movement of dislocations and hence increasing the brittleness of the metal. Theoretically, it should be possible to reduce the friction forces opposing the movement of dislocations:

- either by reducing the number of interstitial atoms such as carbon and nitrogen;

- or by reducing the number of defect clusters, this being done by stepping up the initial number of dislocations (it will be recalled that their density depends on the initial microstructure of the steel, its degree of cold-working, etc.), which, as Eyre has shown (Ref. 39), can absorb defects and hence reduce them in number;

- there is also a third method which accounts for the attraction of high irradiation temperatures (and hence vessel operating temperatures) and that of post-irradiation annealing; this is to increase the temperature, which causes modifications in numerous processes, creates vacancies of thermal origin, renders the defect clusters unstable or prevents their formation, etc.

A brief recapitulation should be made of a few basic concepts relating to the brittle fracture of body-centred cubic metals, in particular iron.

Some of these theories are based on an empirical relation suggested by Hall (1951) and Petch (1953) connecting the lower yield point σ_y with the average grain diameter (2d)

$$\sigma_y = \sigma_i + k_y \, (d)^{-1/2}$$

Although there are still some doubts as to the physical significance of σ_i and k_y, considerable use is made of this equation, which can be interpreted as follows:

σ_i can be regarded as the friction stress of the lattice, i.e., it constitutes a means of measuring the resistance of the lattice to the movement of the dislocations (it will be recalled that σ_i has a high value in the case of ceramic materials, which are by virtue of this fact brittle).

In body-centred cubic metals, σ_i is very temperature-dependent, which provides an indication of a corresponding relation between brittleness and temperature.

It should also be pointed out that σ_i sometimes increases to a marked extent with the content of alloying elements and impurities, whether in solid solution or in precipitate form. It was found experimentally, for instance, that any increase of 0.01% in the carbon or nitrogen in solution in the iron raises σ_i by about 4.6 kg/mm^2. Phenomena such as strain-ageing or quenching produce the same effect.

With regard to irradiation, Churchman, Mogford and Cottrell (Ref. 40) found that this increased the transition temperature of a steel by 60°C for an increase of about 25,000 psi (17.5 kg/mm^2) in σ_i.

The other term in the equation of Hall and Petch, k_y, has frequently been regarded as a measure of the stress necessary to unpin the dislocations. It is found to be equal to $\sigma_d \, l^{1/2}$ where σ_d is the stress required to remove a dislocation from its atmosphere and l the distance between the stress concentration point and the nearest dislocation source.

According to Petch, k_y is not temperature-dependent.

In addition, it has been found to be virtually insensitive to irradiation up to an integrated dose of $1 \cdot 10^{19}$ n/cm^2 (Refs. 41 and 42), beyond which dose it drops slightly. This would imply that up to this dose $\Delta\sigma_y = \Delta\sigma_i$ at a rough approximation (it is possible that above $1 \cdot 10^{19}$ n/cm^2 this no longer applies so strictly).

There are several experimental results now in existence which indicate a linear correlation between the variations in Δt, σ_y and $\Delta\sigma_i$.

Wessell (Ref. 43) obtained experimental values for $\Delta\sigma_y$ and Δt on irradiated samples of A-302-B steels which show a linear correlation between Δt and $\Delta\sigma_i$.

Nichols and Harries (Ref. 44) detected a linear correlation between $\Delta\sigma_y$ and Δt, while Harries (Ref. 45) found that $\Delta\sigma_y$ and Δt are both proportional to the square root of the integrated dose for a steel containing 1% Cr and 0.5% Mo, irradiated at 250°C up to $2.5 \cdot 10^{19}$ n/cm^2 ($>$ 1 MeV). This had in fact been predicted theoretically by Cottrell (Ref. 46) in 1958, who proposed the following for notched steel bars:

$$\Delta t = 7 \cdot 10^4 \, \Delta\sigma_i$$

($\Delta\sigma_i$ in shearing modulus units

Δt in °K)

As is reported by Shure (Ref. 47), confirmation can be found for the correlations quoted above by applying Cottrell's equation to the "sensitive" and "insensitive" heats of A-302-B steel studied by Carpenter (Ref. 10). Comparison of the calculated shifts in the transition temperature and those obtained experimentally shows values of 246°F for 240 and 55°F for 45-100 respectively. It therefore appears that there is good reason for thinking that a reduction in the transition temperature shift can be effected by cutting down the frictional forces.

Practical Application - the Action of Nitrogen

Proceeding along the same lines, various considerations on the behaviour of interstitial atoms prompted us, back in 1963, to study the part played by nitrogen in irradiation damage to iron and steels (Ref. 48).

One of the chapters in this study concerns the shift in the impact transition temperature of steels containing nitrogen to which were added specific quantities of elements known for their varying affinity for nitrogen and capable of forming stable combinations with it (e.g., aluminium, titanium, etc.). The main results, which are to be found in various publications (Refs. 49, 50, 51 and 52), are summarized below.

In the first study (Ref. 49) three iron alloys were prepared with an almost identical carbon content (0.0070 to 0.0086%) and nitrogen contents of the order of 0.020%. However, the killing elements used in the three alloys differed (Si, Al, Ti), two of them, Ti and Al, being added in sufficient quantity to combine with all the nitrogen present (and in the case of the titanium with the carbon as well). The assays of these three alloys are given in the table below. Their ferritic grain is almost identical and varies by 1000 to 1500 grains per mm^2.

Table 11

Composition of the Three Alloys (wt.%)

	A Fe-Ti-N Alloy	B Fe-Si-N Alloy	C Fe-Al-N Alloy
C	0.0086	0.0075	0.0070
N total	0.0022 ± 0.0002	0.0255	0.0175
N precip.	0.0021	-	-
S	0.0066	0.0070	0.0084
P	0.0100	0.0110	0.0109
Si	0.0153	0.0580	0.0130
Ti	0.042	-	0.0052
Al	0.0007	0.0008	0.104
V	0.0001	0.0001	-
As	0.0380	0.0225	0.0200
Cu	0.0209	-	0.0350
Mn	0.0103	0.0120	0.0340

The shifts in the transition temperature (in this case the temperature at which half the maximum energy is obtained) are given below for an integrated dose of 1.4 . 10^{19} n/cm^2 and an irradiation temperature of 50°C.

Table 12

Condition	Material		
	A (Fe-Ti-N)	B (Fe-Si-N)	C (Fe-Al-N)
Non-irradiated Irradiated	-59°C + 3°C	+ 20°C +180°C	-49°C +17°C
t (°C)	62	160	66

The impact test samples are modified Charpy bars (5 • 10 • 55 mm). The following points emerge:

- in the non-irradiated state alloy B (containing silicon) is more brittle than the other two;

- in the irradiated state alloy B undergoes an increase in the transition temperature of 160°C, as opposed to 62°C for A and 66°C for C (in other words, almost three times that of A and C);

- since the Δt values for A and C are virtually identical, it might be thought that the difference in comparison with alloy B is due to the nitrogen, which is entirely combined in these two alloys. In actual fact the aluminium only combines with the nitrogen, while the titanium can combine with both nitrogen and carbon, generally forming carbonitrides. The similarity between the Δt values would seem to indicate that carbon has no major effect on embrittlement.

However, measurement in the shifts in the transition curves for low neutron doses, e.g., 2 • 10^{18} n/cm^2, reveals a certain difference in the damage, the titanium-containing iron alloy being only slightly affected, while the iron alloy containing aluminium shows a Δ t of 45°C (these two alloys are different from those listed in the table above).

It would therefore appear that the damage kinetics vary depending on the nature of the nitride-forming element, at least in the initial phase. This would appear to be confirmed by the results of impact strength recovery tests performed on Fe-Ti-N and Fe-Al-N, in which the properties of the first alloy were recovered almost entirely after 200 hours' annealing at 300°C, while after 300 hours at 300°C the second still showed a Δ t of about 20°C.

To sum up, it may be concluded that the free nitrogen is to a large extent responsible for the irradiation damage in these alloys and that it would be advisable to block this nitrogen fraction in the form of a stable nitride, at the same time bearing in mind that the nature of this nitride probably also has an effect, apparently secondary, on the reduction of the irradiation damage. If this effect, which is known to exist in iron alloys, were found to occur in steels also, this would represent an important means of improving their irradiation behaviour. Tests on steels are now under way.

CONCLUSIONS

In addition to the possible courses proposed on page 15 of this article, the following suggestions might also be worth considering:

(a) to block the interstitials, and in particular nitrogen, so as to reduce the harmful effect of the free nitrogen on the Δ t value; if the steel contained carbon, the same procedure would be applied provided that this element retained the effect on the strength of the steel which it is known to have;

(b) to reduce the number of irradiation defects or to render them unstable by using steels whose structure forms the seat of a large number of dislocations, which would no doubt be tantamount to corroboration of conclusion 5 on page 16 to the effect that quenched and tempered steel should be developed.

The points which emerge from this study can be formulated as follows:

On the basis of theoretical considerations and in the light of the experimental data available at the present time, the ideal non-austenitic steel for use in the construction of the reactor vessels would appear to be a high-purity (residuals and gases), fine-grain, killed, quenched and tempered steel in which the nitrogen is combined in the form of stable nitrides and whose composition, probably of the Ni-Cr-Mo type, is designed to allow for the specific influences described above. It would also have to be easily recoverable by "defect-annealing" without being subject to thermal embrittlement.

The impact strength in the non-irradiated state would be high and its transition temperature low, in which state it would offer the maximum resistance to all types of embrittlement likely to be encountered in the various stages of its fabrication and processing (cutting, forming, welding, stress-relieving).

As has been seen, these requirements could be fulfilled within a relatively short time by means of the additional research indicated.

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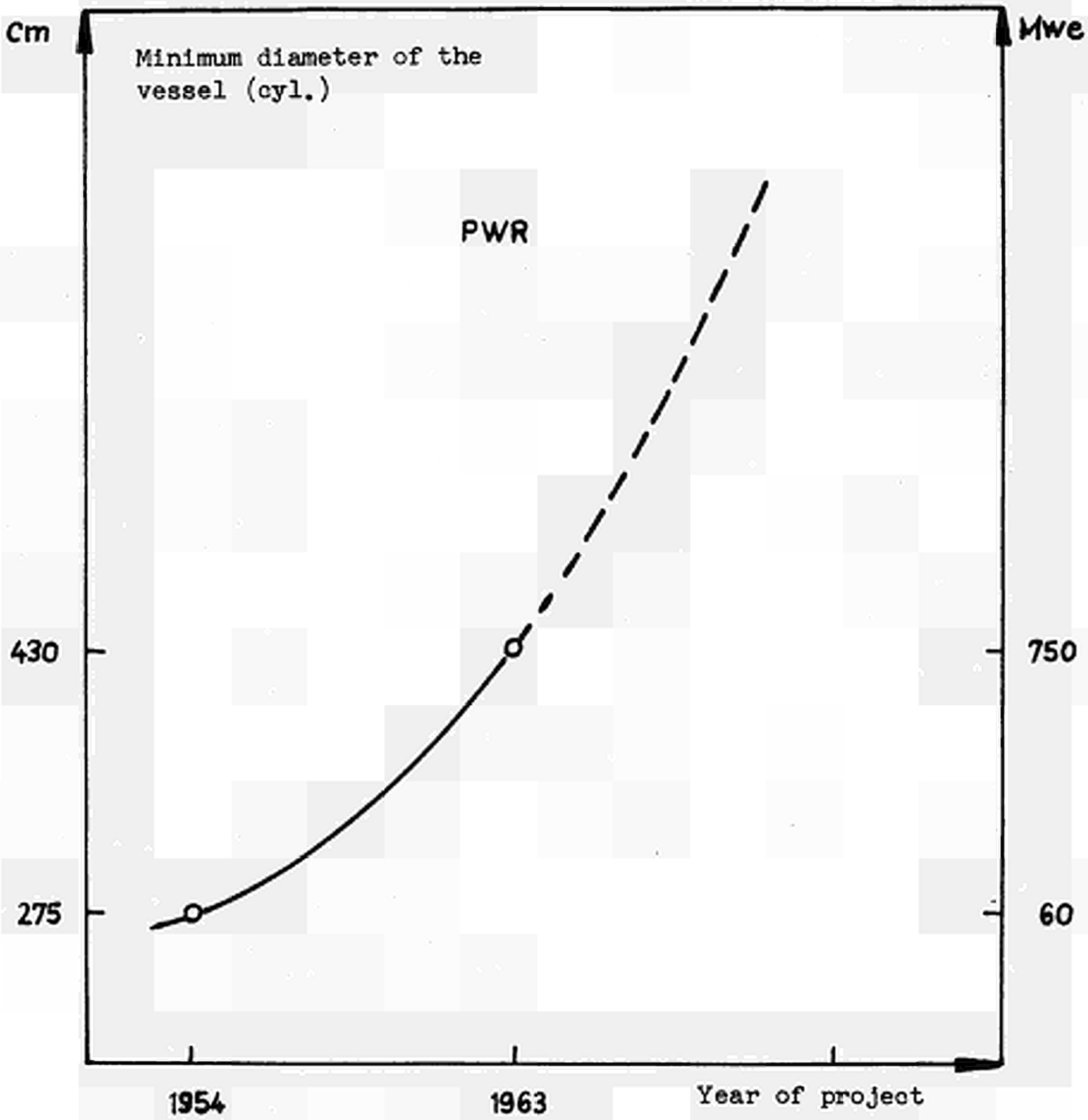
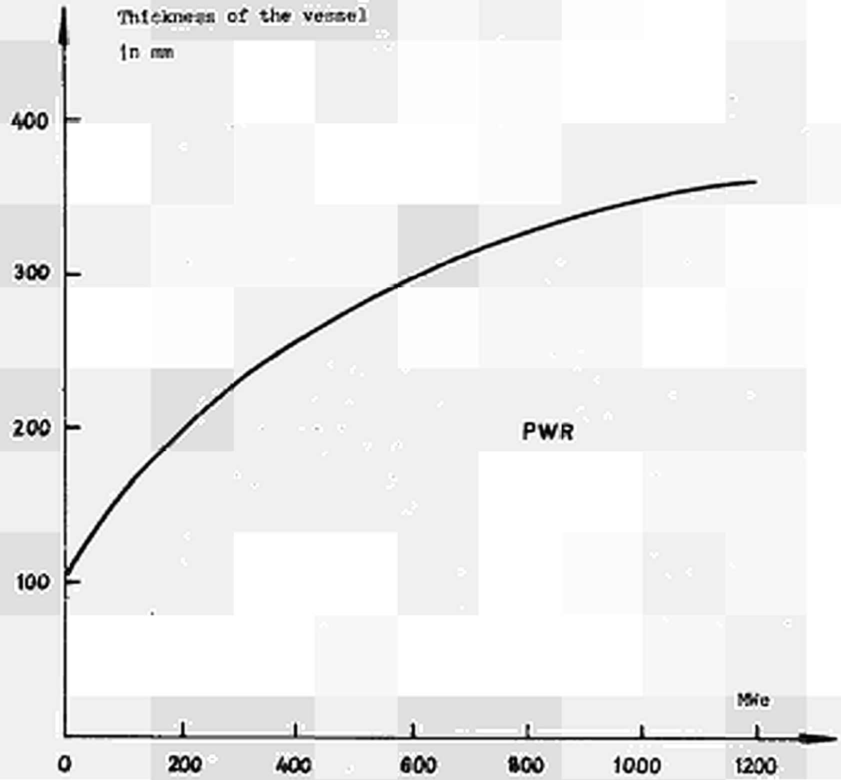
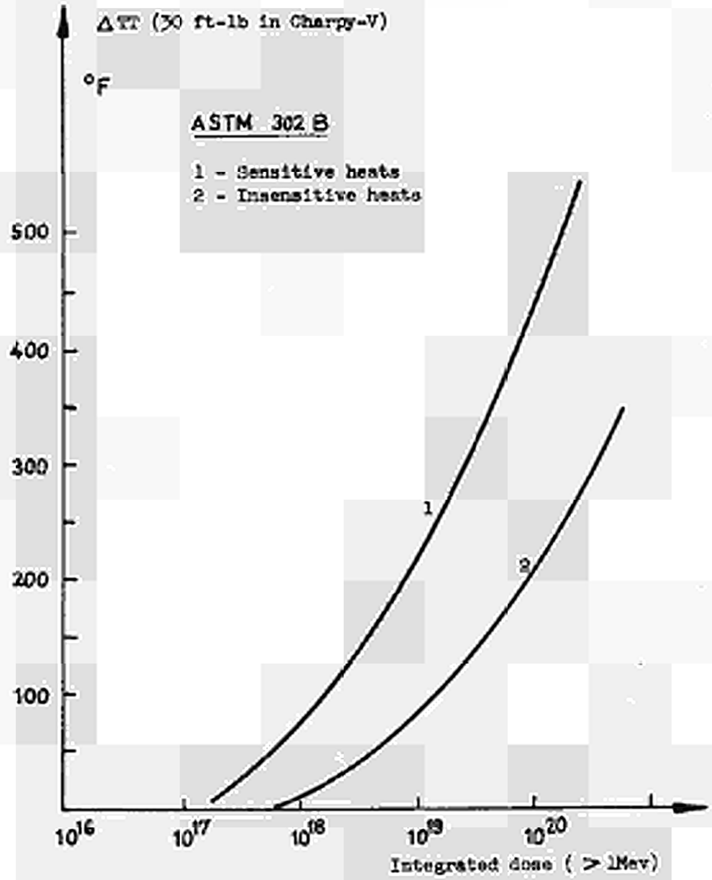


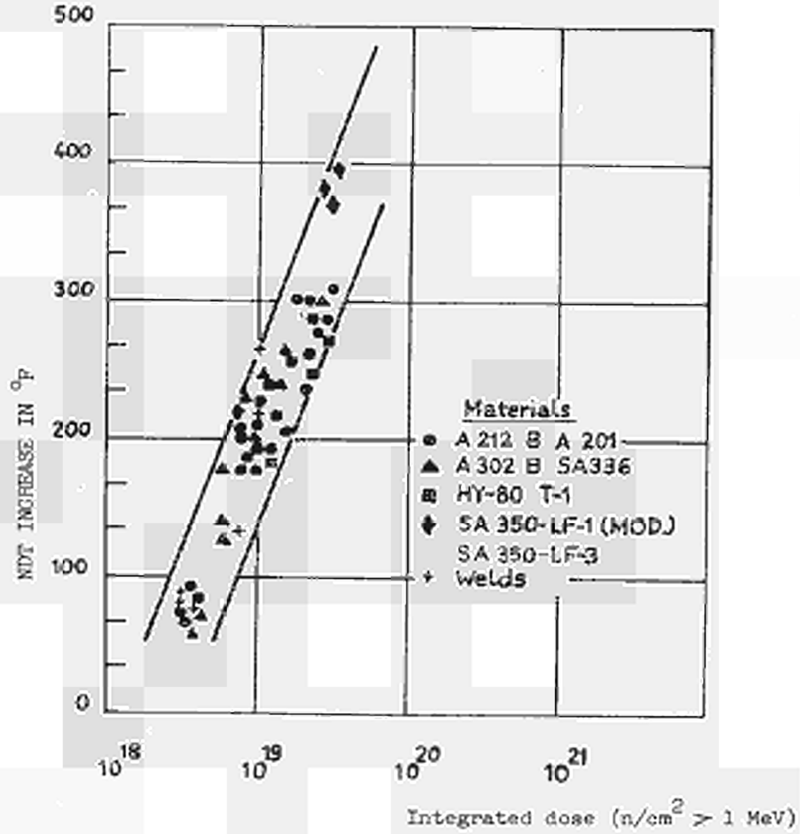
SIZE OF THE VESSEL VERSUS POWER



THICKNESS OF THE VESSEL WALL IN A PWR VERSUS POWER



INFLUENCE OF IRRADIATION ON SEVERAL A-302-B STEEL HEATS



INCREASE IN NDT t° AS A FUNCTION OF INTEGRATED NEUTRON DOSE

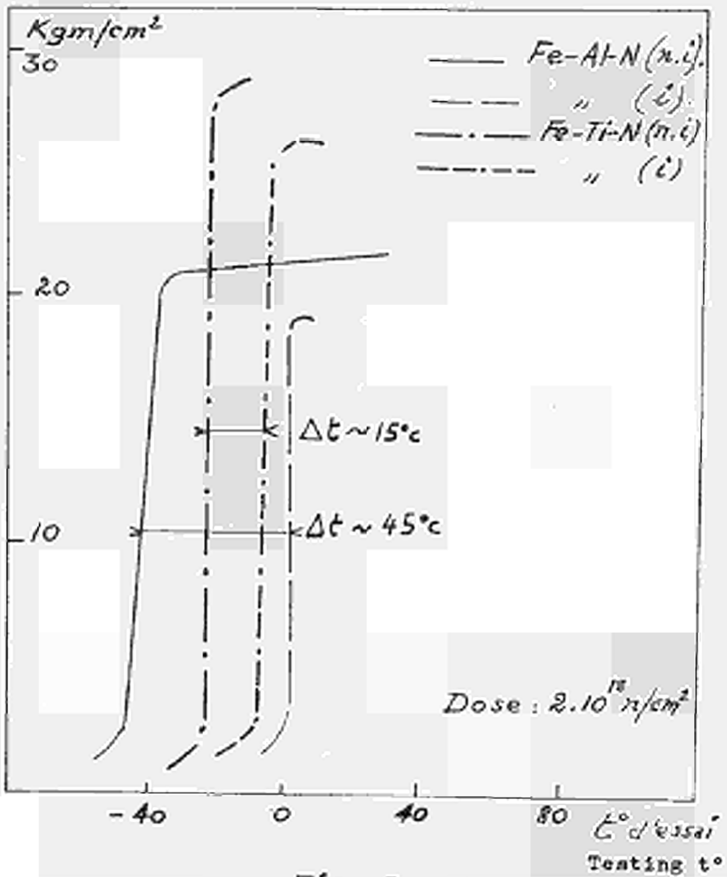


Fig. 5.

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Alfred Nobel

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