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ENERGY
ENGINEERING

ENHANCING THE SURVEILLANCE OF LWR FERRITIC STEEL COMPONENTS

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ABSTRACT: Cost-effective, and safe, management of nuclear power plants requires to insure that fuel, materials and instrumentation will perform in accordance to their design; in this respect, primary loop ferritic steel components of light water reactors are very important, especially the pressure vessel -"never" to fail, costly to replace, and subject to surveillance as specified by Regulation. This paper summarizes current Belgian efforts to enhance reactor pressure vessel surveillance.

Why do we need to improve surveillance ? This question is addressed in terms of four main considerations:

1) Limitations of current methodology to *"index" fracture toughness*
2) Scatter and biases of Regulatory *embrittlement trend curves*
3) Lack of data at desired operation exposures exceeding the range where *incubation of poorly characterized damage mechanisms* may enter into play
4) Possible hidden role of *thermal ageing* effects, not obvious from engineering evaluation of routine tests.

The conceptual approach to answer these concerns combines dislocation-defect damage and micromechanics models (the "software") to the measurement (the "hardware") of instrumented Charpy-V and static tensile load-deflection responses, complemented by limited fracture toughness tests using small specimens; such efficient enhancement of the scope of current commercial surveillance is made possible by the technology of reconstitution of broken C_v-remnants, followed by subsequent remachining to the geometry of interest.

The supporting R&D program calls for verification and further unraveling of the damage modeling formalism by coordinated application of microstructural interrogation techniques associated to isochronal-isothermal anneals; the program plan encompasses the accelerated exposure of selected steels to high neutron fluence in the BR2 reactor and the investigation of trepans cut from two vessels under decommissioning.

This Belgian approach is illustrated by the brief reporting and discussion of recent results.

KEYWORDS: Light Water Reactor steel, surveillance, irradiation embrittlement, thermal ageing, fracture toughness, micromechanics, damage modeling, microstructure, reconstitution.

1.0 INTRODUCTION AND CONTEXT.

This paper addresses the fracture toughness characterization of LWR primary loop ferritic components, with emphasis on the reactor pressure vessel of PWRs.

The *design life* of nuclear power plants is generally considered of the order of 40 years; in some countries, operation *license renewal* after 32 EFPY (effective full power years) is a Regulatory requirement for *plant life extension* (PLEX) while in other countries, like Belgium, *plant life management* (PLIM) per se is the overwhelming consideration: namely, the operation license is granted for unspecified duration, but is subject to Regulatory review every decade- unless some technical surprise arises, calling for plant modifications, component replacement or repair, ... Often, the *economic life* of oldier plants has been less than their design life. Unless plant longevity is encouraged by appropriate action- and given current trends of new plant construction-, a most undesirable decline of world nuclear capacity could occur by the turn of the century [1]. Plant life management is an intricate and multidisciplinary task, calling for serious *cost-benefit* analysis. For instance, it may be pointless to replace a degrading steam generator if Regulatory pressure-temperature limitations for pressure vessel protection during heat-up and cooldown should soon become too stringent (thus expensive); in this example, the situation would be much improved if the overconservative assumption of a vessel quarter thickness flaw was relaxed, or if the fracture toughness at beltline of the exposed vessel could be shown to be superior to current estimates; on another hand, one cannot afford to be penalized either, should the toughness later be found worse than anticipated. In other words, knowledge and accuracy are the relevant considerations.

Fracture toughness requirements for protection against vessel failure [2]-[5] impose to monitor the evolution of mechanical properties by means of an adequate *surveillance program* [6][7]. This is accomplished by exposing, near the vessel, experimental capsules containing test specimens representative of the vessel beltline materials (base metal, weld, heat-affected zone) as well as neutron dosimeters and temperature controls. The irradiation of these capsules is somewhat accelerated with respect to the vessel, generally displaying a "lead factor" of 2 to 5, and thus allowing to anticipate the vessel toughness. In Belgium, the testing of the surveillance capsules of the seven PWR plants currently in operation and the Regulatory-based evaluation are done respectively at SCK-CEN and at Tractebel.

Commercial surveillance in Belgium has so far essentially relied on the Charpy-V notch impact test and, for the unirradiated condition, on the Pellini drop weight test. In the transition temperature range between brittle and ductile fracture regimes, these data are used to estimate [2] [3] lower bounds for the static initiation fracture toughness K_{Ic} , for the crack arrest toughness K_{Ia} and for the "reference toughness" K_{IR}; this last quantity, used to establish heat-up and cooldown limitations, is the lower bound of K_{Ia} and of the dynamic initiation toughness K_{Id}. Vessel integrity evaluation under accidental loads (e.g. pressurized thermal shock) is based on the K_{Ic} and K_{Ia} curves referred to the appropriate equivalent temperature T-RT_{NDT}, where T is the local metal temperature and RT_{NDT} is a reference temperature:

1) For the unirradiated steel, RT_{NDT}^0 is equal to the drop weight nil ductility temperature NDT, or to TT_{68J}-33°C if the Charpy impact energy at NDT+33°C is lower than 68J; here, TT_{68J} is the temperature at which three C_v specimens have an energy ≥ 68J;

2) For the irradiated steel, $RT_{NDT} = RT_{NDT}^0 + \Delta TT_{41J}$, where TT_{41J} is the temperature at which the average C_v energy-vs-temperature curve crosses the 41 Joule level; Δ means the irradiation-induced shift.

It has been shown that the above, Regulatory methodology of ***fracture toughness indexation*** to the arbitrary 41 Joule level of absorbed C_v-energy does present significant drawbacks [8]-[11]. Furthermore, such indexation, combined to insufficient damage modeling insight, leads to unwarranted ***mismatch and scatter of embrittlement trend curves*** based on "engineering" correlations (e.g. [12]). A brief summary of these findings is given in §4.1 below.

End-of-Design-Life (EODL) neutron fluences for Belgian vessels are generally of the order of 4.5E19 cm^{-2} (>1MeV), but may be larger for some units (up to 8E19 cm^{-2} without low leakage core). To reach an *economic plant life of 50 years or more* would entail vessel exposures exceeding the range where ***incubation of poorly characterized damage mechanisms*** may enter into play.

The assessment ***of Thermal Ageing*** is also an important consideration for long term plant life management. In principle, this can be addressed by testing "thermal surveillance capsules", i.e. capsules exposed to temperature but not to radiation. Two potential difficulties need to be overcome:

1) Such effects can remain hidden if investigated only through routine application and evaluation of the Charpy-V notch impact test.
2) Thermal capsules are usually exposed at a temperature of $\approx$ 300°C, but in some plant locations (vessel head, pressurizer,...), the same steels can be subject to temperatures up to $\approx$ 340°C .

Preliminary work relative to this issue is outlined in §6.0.

For all the above reasons, an **Enhanced Commercial Surveillance Strategy** is being developed in Belgium. Overview of this R&D effort in progress is the focus of this paper.

2.0 ENHANCED COMMERCIAL SURVEILLANCE.

Figure 1 presents a simplified, conceptual block-diagram of "Enhanced Surveillance".

Routine, "classical" surveillance in Belgium entails- for each representative metal type in the capsule- the measurement of absorbed energy, lateral expansion and fracture appearance of 8-10 C_v-impact specimens broken at selected temperatures; this is complemented by one tensile test near operation temperature (300°C).

The <u>first main component</u> of "enhanced" surveillance encompasses the following steps:

1) To systematically evaluate the existing load-deflection (time) traces recorded during the Regulation-imposed Charpy tests
2) To test additional, available tensile specimens at lower temperatures
3) To reconstitute some broken Cv-specimens into new ones or into miniature tensile ones, in order to *extend* the above information and allow construction of the

"***generalized load-temperature diagram***" [8]; a summary is given in §3.0 of how such diagram is used.

Reconstitution technology has been systematically developed at SCK-CEN during the last decade, has been pedigreed, commercially applied and is documented in various publications (e.g.,[13]-[15]).

The second main component of "Enhanced Surveillance" relies on the reconstitution technology to prepare 10-12 additional Charpy-type specimens for precracking and slow bend testing in order to measure the static initiation fracture toughness at a few selected temperatures in the lower-to-mid transition range (< 200 MPa√m). This is combined to the generalized Charpy-V load-temperature diagram for a ***micromechanically-based evaluation of static K_{Jc}, dynamic K_{Jd} and arrest K_{Ia} mean trends and bounds.*** Such evaluation is presently somewhat crude, but will be refined in future by directly referring to the localization and nature of the cleavage initiation sites defined by SEM examination, and by more thoroughly addressing the competition with ductile initiation and tearing in the upper transition range.

The third main component of "Enhanced Surveillance" addresses more specifically the actual development of ***plant-specific embrittlement trend curves***. This does not stand in isolation, but is linked to the generalized load-temperature diagram (first component) and to fracture mechanics testing (second component). Miniature mechanical test techniques are intended only for supplementary test reactor irradiations if deemed necessary (for example to expediently scope the possibility of incubation effects at higher neutron fluences); the focus of this third component is on ***microstructure interrogation in support of damage modeling.*** This is briefly reviewed in §5.0.

The various components of this enhanced surveillance capability are held together by **"Advanced Modeling"**, an engineering-oriented synthesis of dislocation theory, irradiation damage modeling and fracture micromechanics. It is clear that a decision as to which "module(s)" of such developing capability is useful and cost-effective for plant life management will depend on the plant problems and specificities, on the prevailing Regulatory context and on the actual achievements of the underlying research effort.

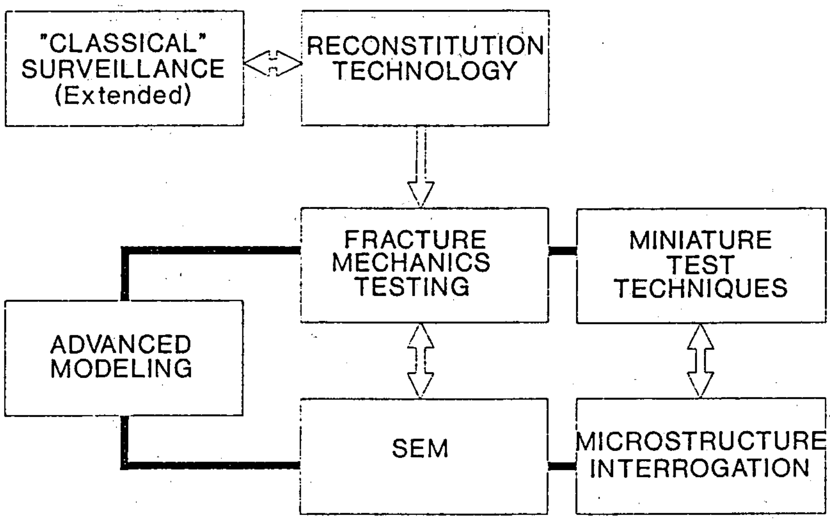

Figure 1. Enhanced Commercial Surveillance

3.0 GENERALIZED LOAD-TEMPERATURE DIAGRAM

The concept of generalized load diagram, described in previous papers ([8], [9], [10]), is primarily useful under stress-controlled fracture conditions, such as, for example, when characterizing the ductile-brittle transition in b.c.c. alloys.

In the present context, the diagram centers first upon the instrumented Charpy-V impact test, as illustrated by Figure 2. On the top part, the four characteristic loads defined by testing standards [7] [16] are plotted in function of test temperature: General Yield F_y, Maximum F_m, Brittle Initiation F_u and Arrest F_a loads, all scaled so as to be consistent with uniaxial tensile yield stresses. The diagram is insensitive to hammer geometry (ASTM vs. DIN tup), to reconstitution, and, in general, to notch orientation; experimental scatter is small for the yield and for the maximum load, which quantify dynamic flow properties (the difference, maximum-yield, correlates well with the Ramsberg-Osgood strain hardening exponent).

The top and bottom part of Figure 2 correlate to one another: namely, the fracture appearance is linked to the load diagram, and this allows to define T_I and T_O, two reference temperatures representative of DBTT, the ductile-brittle transition temperature (these are almost identical for precracked samples, pure iron, binary alloys,..) On average, the steel is brittle at temperatures below T_I, (but may still display some plasticity), while above T_O, it is fully ductile.

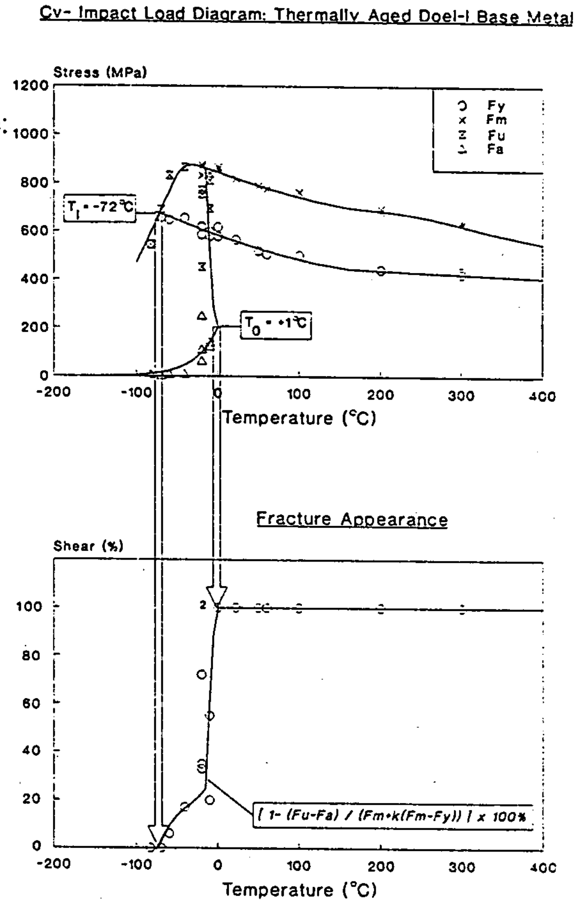

Figure 2. Correlation of Cv- Impact Load Diagram and Fracture Appearance

To good approximation:

> **T_I is adequate for indexation of *dynamic* fracture toughness K_{Id}**
>
> **T_O is adequate for indexation of *crack arrest* fracture toughness K_{Ia}**

The other important facet of the generalized load diagram is that is does contain crucial information in terms of **embrittlement damage mechanisms.** A far reaching example is provided by Figure 3 [10]. Consistent combination of static uniaxial and C_v-notch data (as described in [8]) reveals in this case the presence of a short range, thermally-activated defect complex (represented analytically as a Fleischer tetragonal lattice distorsion[8]). For long range defects, such as copper rich precipitates, only the athermal part of the yield stress is increased (i.e. $\Delta\sigma_y$ is independent on test temperature and strain rate) and there is no detectable change of work hardening; the transition temperature shift ΔTI (= ΔDBTT) is linked to $\Delta\sigma_y$ by the well known Ludwig-Davidenkov diagram- a type of link used (and sometimes abused) by damage modeling experts, dealing most often with medium to high copper alloys (PWRs, 270-300°C). By contrast, short range, thermally activated obstacles affect the temperature-strain rate dependent part of the flow stress: $\Delta\sigma_y$ then depends on temperature and strain rate, left bottom part of Figure 3, and strain hardening is also affected, bottom right.

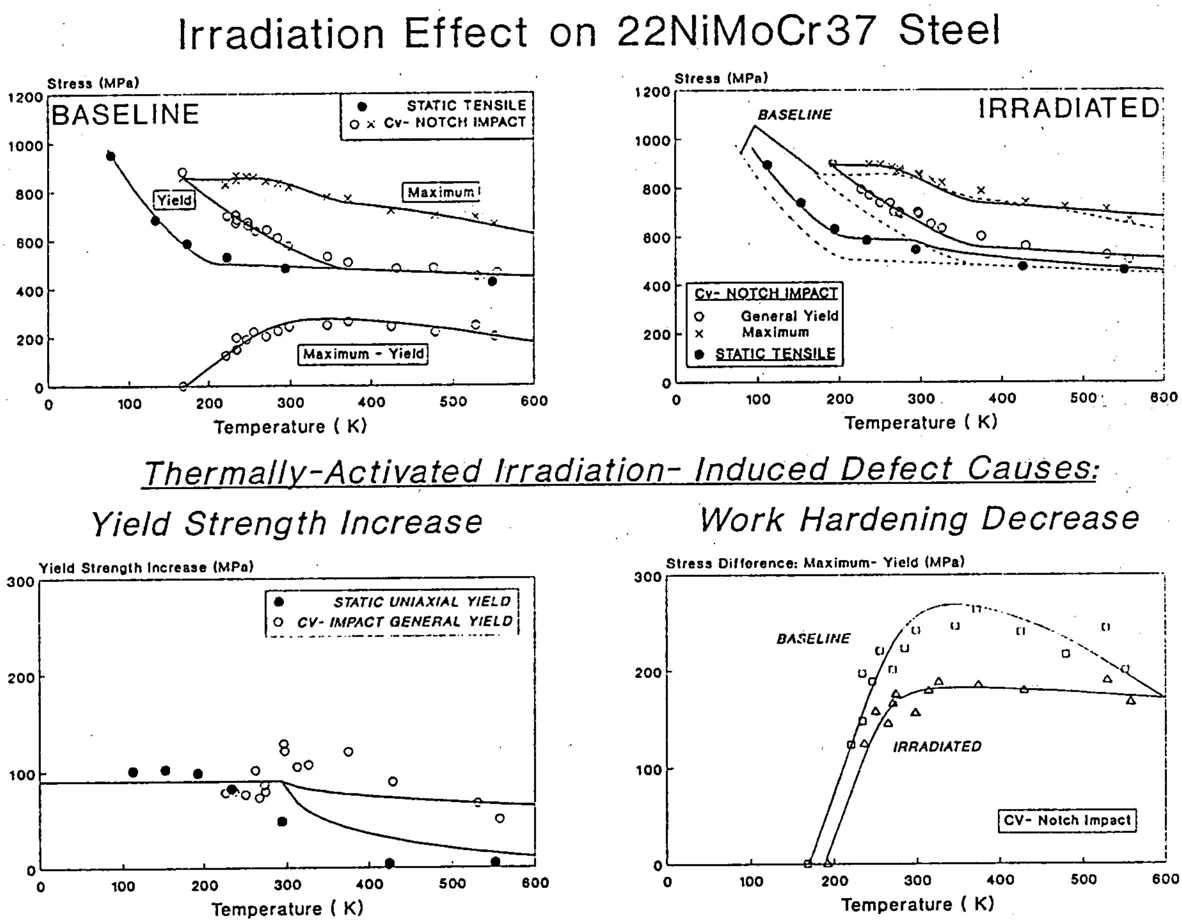

Figure 3. Generalized Load-Temperature Diagram Insights: Damage Mechanisms.

Definition of the average lower shelf ***microscopic cleavage fracture stress*** is another basic information contained in the generalized load diagram; it is derivable from the stress at T_I. As indicated in reference [8], this can be used, in conjunction with the flow properties (σ_y , work hardening) in order to estimate K_{Ic} and K_{Id} ("Modified RKR model"); the estimate can be considerably refined if limited K_{Jc} measurements at one or two temperatures are available; these can be obtained by three point slow bend tests using precracked and side-grooved Charpy size specimens, eventually reconstituted from broken surveillance remnants (e.g., 10 x 10 x 10 mm [17]-[19]).

Finally, the presence of service-induced non-hardening embrittlement (see §5.1) can be unravelled and its importance estimated : this is correlated to any eventual decrease of the stress at T_I.

4.0 FRACTURE TOUGHNESS AND MICROMECHANICAL MODELING

4.1 Background

The Regulatory methodology of fracture toughness indexation to the C_v-notch impact test (§1.0) presents two major drawbacks:

1) Neglecting the influence of irradiation on the *strain rate sensitivity* of the steel can cause *unconservative biases* for the K_{Ic} shift

2)The use of TT_{41J} as *indexation temperature* for the toughness shift can cause *(over)conservative biases* for some steels, but not for others [9].

Micromechanical modeling predicts that the decrease of strain rate sensitivity associated to irradiation strengthening reduces the difference between the lower bounds K_{Ic} and K_{Id} curves; eventually, if embrittlement is large enough, these bounds tend to coincide, in contradiction with Regulation. An example is shown by Figure 4 [20]. Here, the K_{Jc} shift does (significantly) exceed the K_{Jd} shift and the C_v-shift (indexed at 28 J, see below).

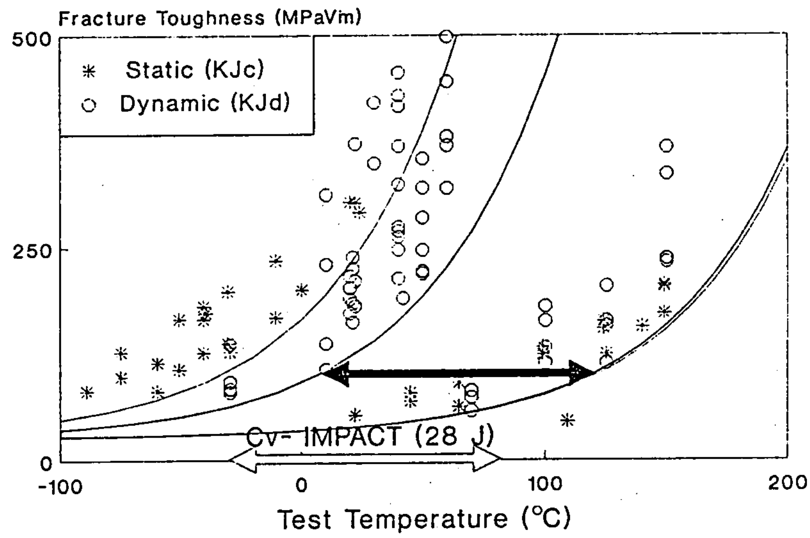

Figure 4. Irradiation reduces strain rate sensitivity, causing lower bound K_{Jc} to reach lower bound K_{Jd}.

One must indeed expect [8] the K_{Id} and K_{Ia} shifts to correlate with the C_v-impact shift, because large strain rates are involved in all cases, and notch sharpness or size effects do not affect much the *shift*, especially if the more physically grounded Charpy transition temperatures T_I and T_O (§3.0) are used. It is thus not surprising that a number of experiments show larger KJc shifts than the Charpy ones. For certain "*outlier*" steels however, the 41 J. C_v-shift can be strongly biased to such extent that it may equal or even exceed the K_{Jc} shift; the basic reasons for such distorsion has been thoroughly explained [8][9] in terms of the load-temperature diagram and of the energy fractions which can be associated to the various physical processes developing during the specimen deformation. The anomaly does essentially go away if the C_v-shift is defined (still conservatively) on basis of the more physical temperatures T_I and T_O. Indexation to TT_{28J} gives usually similar shifts as indexation to T_I, and also, in general, one ly similarfinds that $\Delta TT_I \approx \Delta TT_O$. Finally, the 50% fracture appearance transition temperature FATT provides a further reasonable index, albeit sometimes too conservative (but never the reverse). A striking example of outlier behavior, extensively examined elsewhere [9], is illustrated by Figure 5. Here, two A302-B plates of very similar chemistry, differing only by their prior austenite grain size (due to different austenitization temperatures), are seen to display *drastically different 41J. C_v-shifts,* while the *FATT-shifts* are consistently *similar* within scatter (note that the irradiation temperature for the Yankee/BR3 surveillance plate is in the range 274-282°C, somewhat less than for the ASTM reference plate). It is defended that the *fracture toughness shifts of both plates* is *similar* [9]. This will be *directly demonstrated* by the sampling and testing of the BR3 vessel plate (§4.4): this plate is comparable to the Yankee/BR3 surveillance plate, except for the fact that it has been modified by nickel addition; it has also been contended -at further odds with Regulatory Guide views [12]- that the higher nickel content has negligible influence on the toughness shift.

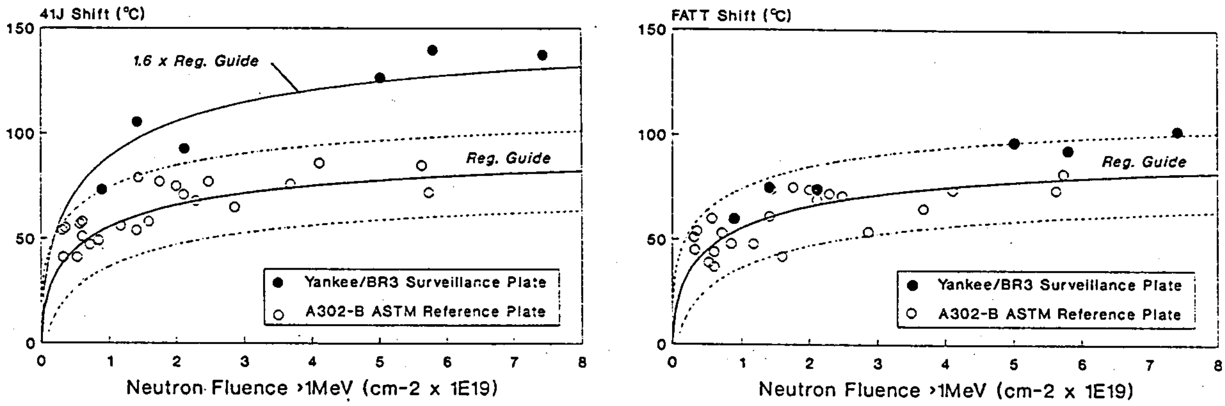

Figure 5. Outlier Behaviour of Yankee/BR3 Surveillance Plate

Toughness indexation aberrations causing apparent outlier embrittlement behavior are not the rule, but neither are they seldom. Such aberrations- if not always as dramatic as in the Yankee/BR3 case - are sufficiently frequent to significantly contribute to the ***mismatch and scatter of embrittlement trend curves*** based on current engineering concepts and correlations. The indexation inadequacies do compound with insufficient consideration of state-of-the-art irradiation damage modeling insight so that the pernicious net effects on Regulatory embrittlement trend curves include [9]: *mean curves* too high, *margins* too high, *chemistry factors* distorted, *neutron fluence dependency* too steep in the range 1-4 E19 cm-² (>1MeV).

4.2 Investigation of HSSI High Copper Weld 73W (In cooperation with ORNL & VTT)

This cooperative venture is a "*benchmarking*" exercise taking advantage of the fact that the high copper weld 73W has been extensively characterized at ORNL, in both the baseline and irradiated conditions (e.g. [21]-[23]): this includes C_v, tensile, Pellini drop weight, K_{Ic} (K_{Jc}) (up to 8T-CT, resp. 4T-CT in the baseline, resp.irradiated conditions) and K_{Ia} tests.

The primary joint objective agreed upon by the three laboratories is
"*To demonstrate that small specimens reconstituted from broken remnants can be used to define the average shape and irradiation-induced shift of K_{Jc}-temperature curve, with same*

accuracy as reached by ORNL using large compact specimens. Statistical effects are to be experimentally assessed at one temperature selected within transition range".

In addition, SCK-CEN is applying its "Enhanced Surveillance Strategy", as outlined in this paper.

The inventory of unirradiated broken 4T-CT, 2T-CT and arrest specimens transferred to SCK-CEN allows to reconstitute a sufficiently large number of small specimens to complete such program- including one irradiation of 18 reconstituted C_v-samples in BR2. Unfortunately, no remnant irradiated at ORNL has yet been shipped.

Some initial, most promising results of this effort are summarized on Figure 6..

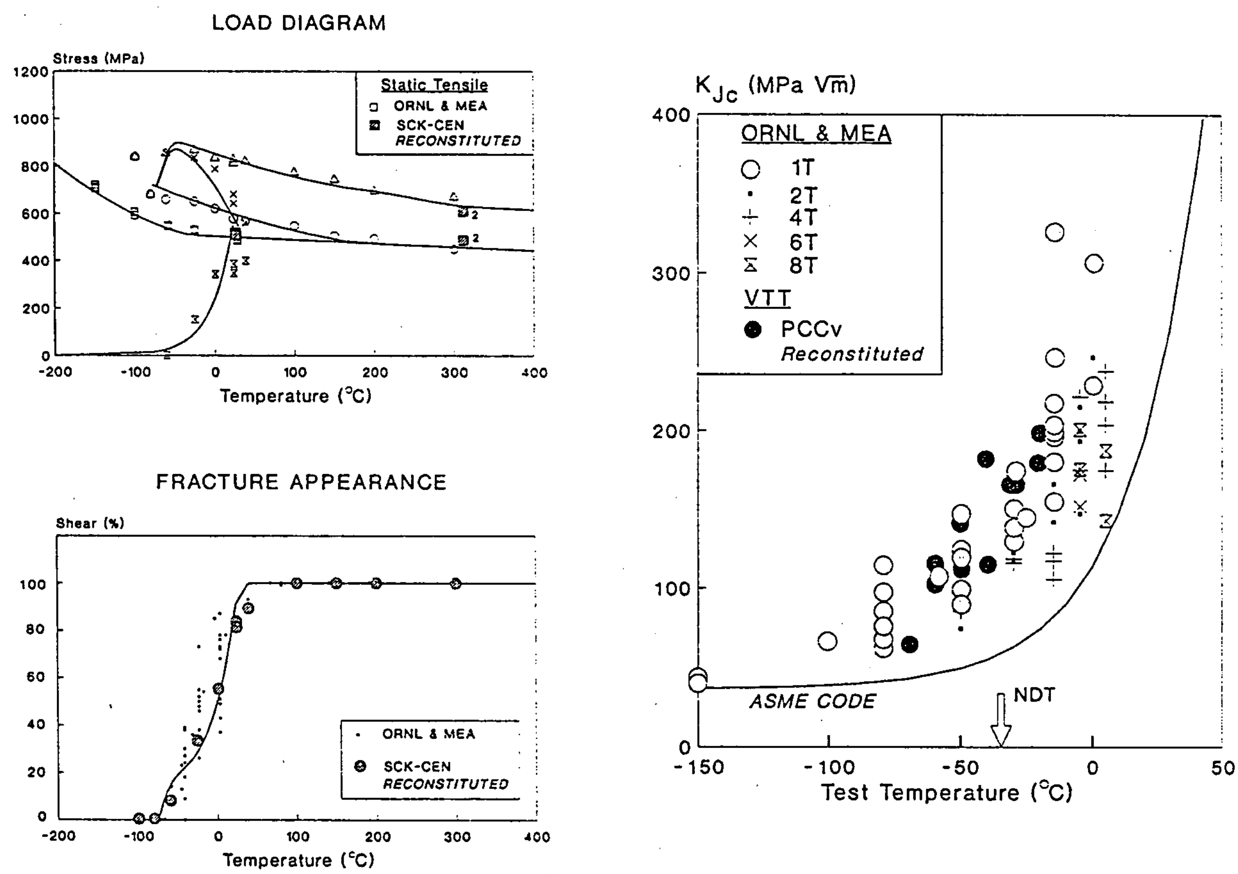

Figure 6. Fracture Toughness Using Small Specimens:
Initial Results of ORNL/VTT/SCK-CEN Demonstration Project for HSSI Weld 73W

The left-hand side of the Figure displays the generalized load-temperature diagram obtained at SCK-CEN. All new specimens have been reconstituted, including the tensile specimens (in good agreement with the original ones for the same orientation); the characteristic C_v-loads are defined as on Figure 2. Correlation of the fracture appearance with the loads is excellent, leading also to good consistency with the ORNL/MEA shear data. The micromechanical modeling evaluation is in progress.

The right-hand side of Figure 6 shows the initial three-point slow bend test results obtained at VTT by means of reconstituted precracked Charpy's from the same stock ; the measurements have been size-corrected to 25 mm thickness and agree well with the original 1T results, including in presence of some ductile crack extension (above NDT). Wallin [24] has successfully applied his "master curve" approach to all these K_{Jc} data.

4.3 Investigation of Trepans from Chooz-A Vessel (In cooperation with EdF)

The Belgian participation to this important effort focuses upon the application of the "Enhanced Surveillance Strategy" outlined in this paper. The trepans are being prepared at this time. Initial damage modeling evaluation of the Chooz-A surveillance data is illustrated by Figure 7 [9]; the damage mechanisms labelled 2 and 2A on this Figure are briefly discussed in § 5.1.

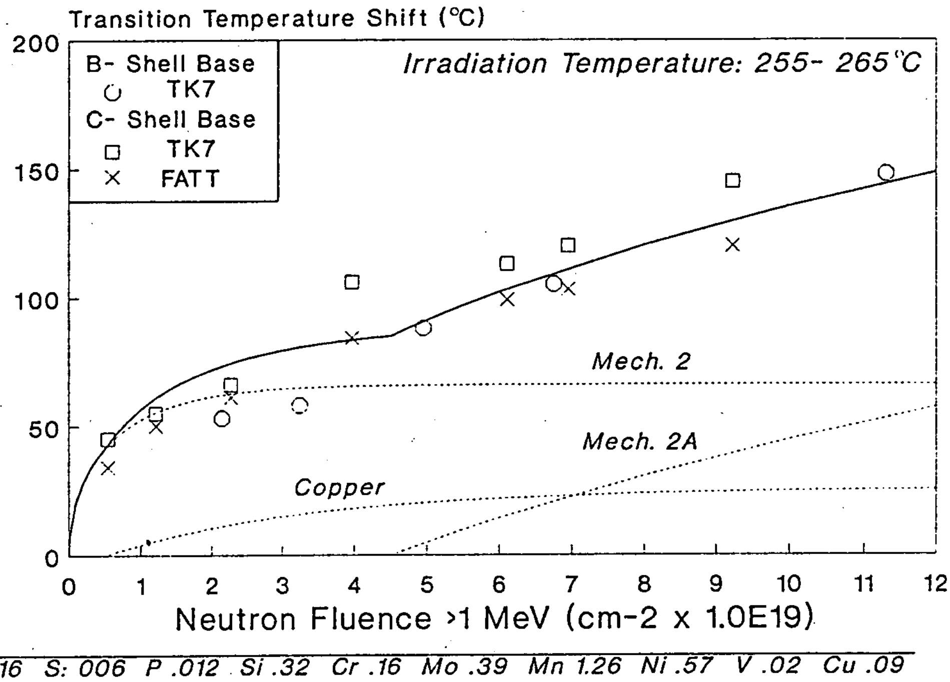

Figure 7. Embrittlement of Chooz-A Surveillance Base Metal Is Well Predicted by Model

4.4 Mechanical Testing and Microstructural Characterization of Pressure Vessel at Decommissioned Belgian BR3 Plant.

The Belgian BR3 plant is presently under decommissioning and SCK-CEN has decided to sample its reactor pressure vessel, in order to proceed to its "Mechanical Testing and Advanced Microstructural Characterization". Investigation of this vessel is important for a number of reasons [25], but what must be emphasized in the present context is that the BR3 plate (similar to the Yankee Rowe nickel-modified lower shell plate) is an outstanding "*Fracture Toughness Indexation Outlier*"(§4.1). This is important insofar as the other ongoing investigations of correlations between fracture toughness and Charpy-V shifts encompass more "regular" steels, likely to display excess K_{Jc} shift: the detrimental role of "outliers" (§4.1) may be missed and it cannot be ruled-out that the current "41J. recipe" be continued, amended with some Regulatory "penalty".

The BR3 vessel sampling and testing program can be synthetized as follows:

Phase I: 1995-1996 *"Blind Trepans"*

I/1 - ***Fracture Toughness from Small Specimen Testing***
Cv, Tensile, PCCv (Static and Dynamic), 0.5T-CT

I/2 - ***Microstructural Characterization***
TEM, FEGSTEM, SANS, Positron Annihilation, Internal Friction, Mossbauer Spectroscopy, ASAXS

I/3 - ***Test Reactor Irradiation*** (BR2: §5.3) of Thermally Aged specimens extracted near nozzle elevation

Phase II: 1997-1998

Large Specimen Fracture Toughness Testing with emphasis on Irradiated KIc and KIa

The Phase I BR3 vessel sampling is contracted to take place in January 1995. The geometrical specification for the blind trepans is sketched on Fig. 8.

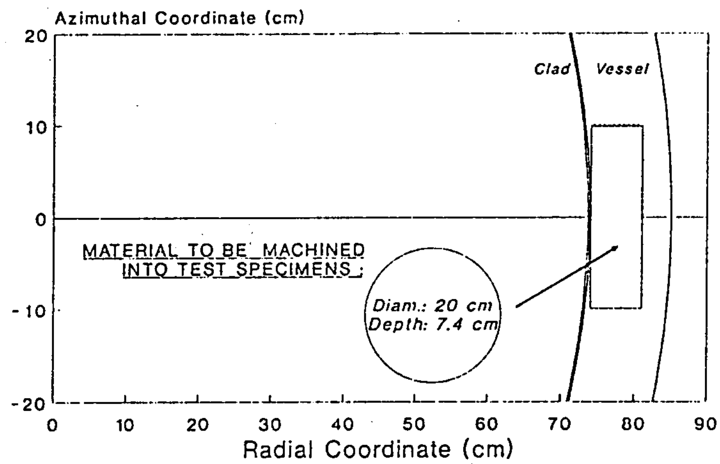

Figure 8. BR3 Vessel Sampling: Blind Trepan Specification

4.5 Micromechanical Modeling of Ductile Fracture.

The approach combines the data obtained from the simple tensile test with finite element calculations and takes into account the micromechanisms of deformation and fracture.

The ductile fracture is a cavitation process. Physically, it consists of three phases: nucleation, growth and coalescence of voids around second phase particles. Its damage kinetics can be schematically formulated by:

$$\dot{D} = f\,(\sigma_{ij}\,,\, d\varepsilon'_{ij}\,,\, \alpha_k)$$

where $\dot{D}$ is the damage rate, σ_{ij} are the stresses, $d\varepsilon'_{ij}$ are the plastic strain rates and α_k are the parameters of the model. The latter are derived from simple tensile tests. Two ductile models have been successfully used to describe the ductile fracture of reactor pressure vessel steels, i.e. the Rice and Tracey cavity growth model and the damage work model [26],[27].

One large advantage of this kind of micromechanical approach is the possibility to incorporate the radiation damage. This can be done by introducing other variables into the model that epresent the radiation effect. Subsequently, no sophisticated tests are required: the tensile test suffices [28].

The quantities derived from these calculations are local, which allows a better understanding of some experimental results: size effect, geometry effect. Furthermore, statistical effects can be easily taken into account [29], due to the local nature of the models.

5.0 INCUBATION AND KINETICS OF RPV DAMAGE MECHANISMS

5.1 Damage Modeling Revisited: Highlights

Until recently, the so-called "**Two-component Approximation**" to RPV steel embrittlement seems to have reflected the predominant views among the more fundamentally-oriented experts such as the ones found at IGRDM: International Group on Radiation Damage Mechanisms. This approximation- a potentially considerable improvement as compared to empirical Regulatory formulations (e.g. [12])- treats embrittlement as due to the *linear superposition* of the strengthening effect of two main types of obstacles to dislocation mobility:

1) *Hardening related to Precipitation of Impurities*, such as copper and phosphorus;
2) *Hardening stemming from "Matrix Damage"*, a term referring to a more elusive contribution of defects induced by displacement cascades, and generally assumed to consist of *stable nanovoids* in the lattice within the grains or laths.

This approximation neglects a third component, "**Non-hardening Embrittlement**" associated to the segregation of solutes at the grain boundaries: the resulting decohesion of the boundaries can trigger intergranular fracture, i.e. whenever the "critical intergranular fracture stress" is less than, or has decreased, upon irradiation, below the microscopic cleavage fracture stress -above a given *incubation fluence* [30]; such decrease causes an increase of the ductile-brittle transition temperature (DBTT), even if the yield strength remains constant. This effect is also reflected by the generalized load-temperature diagram.

Even in absence of non-hardening embrittlement, it is becoming rather clear that the above "two-component" model is too simplistic. It can be contended that instead of nanovoids, "*Matrix Damage*" entails *two distinct, rather intricate types of defect complexes*, probably involving small vacancy clusters stabilized by precipitates- similar in this respect to copper rich complexes. These defects, whose strengthening effect is believed quadratically superposable, are in synergistic interaction with one another, and their kinetics seems to be influenced by competition between austenitizers (nickel) and carbide/nitride formers (Cr, V, ...)[31].

The *most unstable complexes*, labelled "Defect mechanism 2" (according to a nomenclature by D. Pachur [32]) are thermally activated short range obstacles affecting the generalized load-temperature diagram in the manner illustrated by Figure 3. Such defects, strongly influenced by the irradiation temperature and possibly by the dose rate, lead to what we can conceptually describe as "**Strain Hardening Embrittlement**" [31]: in particular, they affect the strain hardening exponent and the plastic contribution to the Vickers hardness response so that the often quoted literature relationship $\Delta\sigma_y = C.\Delta H$ between yield stress increase and hardness increase is no longer valid; an example is given by Figure 9.

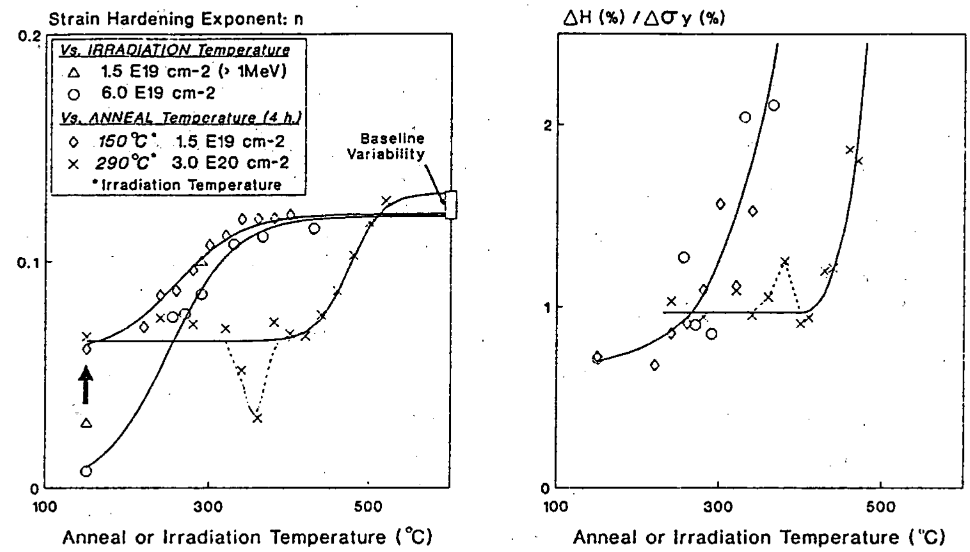

Figure 9. Exposure and Anneal of Plate HSST-03 at Varying Temperature and Accelerated Neutron Fluences Allows Unravelling of Irradiation "Matrix Damage" Components
(Re-evaluation of Pachur's Pioneering 1980 Experiments [32])

This figure is consistent with an anneal kinetics of order >1 and with the finding [ ] that these defects are so unstable as to induce strain ageing recovery for irradiated steels even at room temperature [10]. Finally, it is important to stress that these defects also cause a strong variation of the ratio $\Delta TT_{41J}/\Delta\sigma_y$ with irradiation temperature (See [9], Figure 12.d) and with neutron fluence below 1E19 cm-² (>1MeV).

The other complexes forming "Matrix Damage" are significantly more stable and are labelled "<u>Defect mechanism 2A</u>". They display an ***incubation fluence*** function of the alloy composition, slightly variable with the irradiation temperature; they are little or not affected by the dose rate. These complexes play an ***important role in terms of long term plant life management or of life extension.***

To place these highlights in a more complete perspective, it is necessary to briefly comment on the role of the fine copper rich precipitates which tend to govern embrittlement below 4E19 cm-² in many pressure vessel steels with bulk copper content exceeding 0.10%, and operated in the temperature range 270-300°C. By contrast to current Regulatory views, it is growingly evident that copper in solid solution, rather than bulk copper, is the culprit. The solubility limit during post-weld heat treatment of most vessels is of the order of 0.20-0.25%, and the excess in welds with higher bulk content is found, already in the baseline condition, as fcc copper and copper sulphide precipitates, unable to contribute further to the steel strengthening in service. Linde 80 welds as the ones at the BR3 and Yankee Rowe plants offer a good example [9]. An even more interesting situation has been revealed in the case of the low nickel Doel-I and Doel-II pressure vessel welds, irradiated {seven surveillance capsules, showing little neutron fluence dependence from 1E19cm-² up to 4E19 cm-² (>1 MeV)}[11]: here, the embrittlement of low copper (0.13wt%) specimens is essentially the same as for high copper ones (0.33-0.35wt%), even though the soluble content still differs by almost a factor 2. The reason for this paradox has been identified by means of Field

Emission Gun Scanning Transmission Electron Microscopy (FEGSTEM) performed at AEA, Harwell: the *volume fraction* of irradiation-induced copper-containing precipitates is *similar* for both the low copper and high copper welds; only the average compositions are markedly different:

Low Copper Weld: 36%Cu-33%Mn-10%Ni-11%P-5%As

High Copper Weld: 72%Cu-16%Mn- 4%Ni- 2%P-3%As

These welds are the first commercial Belgian materials to which "Enhanced Surveillance" is being systematically applied [11].

5.2 **Microstructure Interrogation Techniques**

As indicated by the previous sections, a more fundamental understanding of embrittlement mechanisms in terms of microstructure changes induced during service is most beneficial, allowing in particular to establish and defend plant-specific fracture toughness trend curves, even in cases of significant outlier behavior as compared to current Regulatory prescriptions. In addition to high resolution analysis (APFIM, FEGSTEM) of fine precipitates, grain boundary segregations, dislocation structure (TEM),etc, a few techniques -of a more integral nature- have the potential -if applied together in a coordinated manner- to help unravel the complexities of the many still ill-understood features of embrittlement, such as "Matrix Damage". A brief survey of the techniques singled-out for development and application at SCK-CEN follows.

Positron Annihilation Spectroscopy (PAS) [33]

The positron annihilation method is considered to be a valuable tool to obtain information about vacancy-type defects in metals. Positron annihilation lifetime measurements on irradiated steel are not evident, due to the induced ^{60}Co activity: cobalt-60 emits two coincident gamma rays which produce false triggers in a classical lifetime setup, giving rise to a very disturbing prompt peak in the lifetime spectrum. At SCK-CEN, a new positron lifetime measuring system has been constructed, based on three photon detectors instead of two; this allows to distinguish between ^{22}Na events (the positron source) and ^{60}Co events, by requiring a triple coincidence to accept counts in the lifetime spectrum: indeed, three photons are present only if we are dealing with a real annihilation of a positron (the start gamma and two 511 keV annihilation gammas). To obtain an acceptable count rate using a triple coincidence requirement, high efficiency BaF_2- scintillators are used.

In collaboration with the RUG-Positron Laboratory (Drs. D. Segers and L. Dorikens-Vanpraet), some survey measurements have been performed on unirradiated steel, deformed and non-deformed, of the Doel IV plant. Isochronal anneal curves have been measured, showing drastic changes in the mean positron lifetimes.
It is planned to perform similar experiments on a variety of irradiated steels relevant to the Belgian program.

Internal Friction (IF) [34]

Mechanical spectroscopy [35] is another valuable technique for the microstructural investigation of pressure vessel steel embrittlement. In cooperation with RUCA (Prof. R. De Batist) and with Ecole Polytechnique Fédérale de Lausanne (Prof. W. Benoit and Dr. R. Schaller), a torsion pendulum was used to subject small rods of square cross-section to a torsional stress. Subsequently, the stress is removed and the resulting free decay -identified by the symbol Q^{-1} (as defined for instance in [36])- is measured as a function of both the temperature and the applied vibration amplitude. The curves thus obtained (example in §6.0) can yield essential information on dislocation structure, precipitations, segregations at grain boundaries and on the interactions between various structural defects; these processes are known to influence embrittlement.

Mossbauer Spectroscopy (MS) [37]

This technique is able to detect changes in the local environment of the iron atoms present in the material under investigation, and thus can be used to study such changes in RPV steels upon neutron irradiation or thermal treatment. The technique has been applied for the monitoring of the matrix depletion in thermally treated, high copper, IAEA reference material JRQ. Comparison of results obtained before and after a reverse temper embrittlement (RTE) treatment revealed that the matrix signal in the Mossbauer spectrum was significantly enhanced in the RTE treated material. This indicates a (partial) depletion of the matrix. Moreover, the characteristics of the Mossbauer spectrum after defragilisation were identical with those obtained for the untreated material; this tracks RPV steel recovery in a convincing way. The same matrix depletion mechanism has also been recently observed by Lipka et.al. [38] in irradiated steels, including its recovery upon thermal annealing. In addition to the monitoring of matrix depletion, MS will probably allow to selectively investigate the stability of iron-containing carbide precipitates under service exposure.

Anomalous Small X-Ray Scattering (ASAXS)

The first SCK-CEN scoping experiments are planned for Spring 1995.

5.3 **Accelerated Irradiations in BR2: 1995 Program Plan Outline.**

An important finding of recent damage modeling work is that the kinetics of the copper precipitation component is so much slown-down by increasing dose rate as to become almost negligible near 1E14 cm-².sec-1 (>1MeV). This is illustrated by Figure 10 [9].

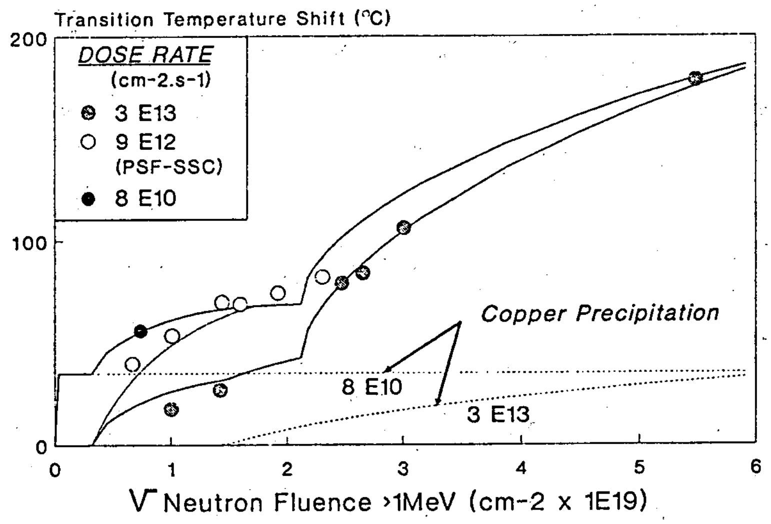

Figure 10. Controlled Test Reactor Experiments Allow to Unravel Dose Rate Effect on RPV Steel Damage Mechanisms (A533-B Plate HSST-03 Exposed at 290°C)

Given this and the discussion in § 5.1, it is clear that exposure at such high dose rate, at two high neutron fluences (respectively of the order of 5E19 and 1E20 cm-²), and at two irradiation temperatures (150°C and 260-270°C), must allow to separate and characterize well the components of "Matrix Damage", i.e. the defect mechanisms 2 and 2A. The pressurized water loop Callisto [39] is particularly suited for such undertaking. A first scoping irradiation at 290°C is presently being prepared, for in-pile insertion within a few weeks. The steels are selected in order to allow relevant comparisons to recently re-evaluated [31] literature work (Plate HSST-03, A302-B Plate YA9 irradiated at the Ford Test Reactor, see below), to a previous BR2 irradiation of a reference steel in the Vestal capsule and in a Belgian surveillance capsule (reference IAEA monitor JRQ) [40] and to other materials under procurement. This will be followed by a series of further accelerated irradiations, to be completed by Summer 1995. Among various steels of interest, it is intended to expose (at 260-270°C and at two fluences) Charpy-V, tensile and microstructure interrogation specimens sampled-out from the thermally-aged base metal at nozzle elevation in BR3 (§4.4); comparison will be made to the results at BR3 vessel beltline for a same fluence (4E19cm-²) as well as to the Yankee/BR3 surveillance A302-B plate results [9]; furthermore, it is planned to simultaneously expose the Yankee Rowe upper and lower shell surrogate plates YA9 (A302-B, coarse grain) and YA1 (nickel-modified, coarse grain) [41], in cooperation with Yankee Atomic Electric Company. Some of these steels will also be irradiated to high fluence at 150°C. The experimental test matrix will incorporate selected isochronal anneals of miniature mechanical and of microstructure interrogation specimens, as part of doctoral and post-doctoral research activities in the frame of ongoing cooperations between SCK-CEN and Belgian Universities.

6.0 THERMAL AGEING OF RPV STEELS.

It has been previously found [11] that thermal ageing at ≈ 300°C after 8 years of operation, i.e. during ≈ 63000 hr., decreases the C_v-upper shelf of the Doel-I,-II welds from ≈140 J to ≈ 115 J; this was accompanied by a small upward -shift of transition temperature. At first glance, i.e. *judging from the conventional Charpy curve*, there was no ageing influence for the simultaneously exposed base metal. Application of the generalized load-temperature approach did however reveal an increase of the characteristic temperature T_I from the baseline value of -100°C to a higher value of -72°C (Figure 2 illustrates the aged condition); this is a shift of 28°C, to be compared with a shift $\Delta T_I = 57°C$ for a similar surveillance capsule exposed to the radiation field: in other words, it was *suggested that, for this low copper base metal, time at temperature induces a K_{Id} shift about half the one expected from irradiation at temperature for comparable time*. The C_v-upper shelf is not affected in this case. Note that this material displays irradiation effects similar to the ones shown on Figure 3, i.e. thermally-activated defects play an important role.

A remarkable confirmation of the significance of thermal ageing for this Doel-I,-II base metal has recently been provided by internal friction studies in progress: see Figure 11 below [34].

The spectrum of internal friction Q^{-1} as function of temperature is measured only after deformation (12-16% in torsion at 250K): the deformation can free dislocations from their pinning points. With reference to the work of A. Munier [36] on JRQ and A533B Class1 steel, we can make a preliminary interpretation of the spectrum relating to the interactions of dislocations with defects.

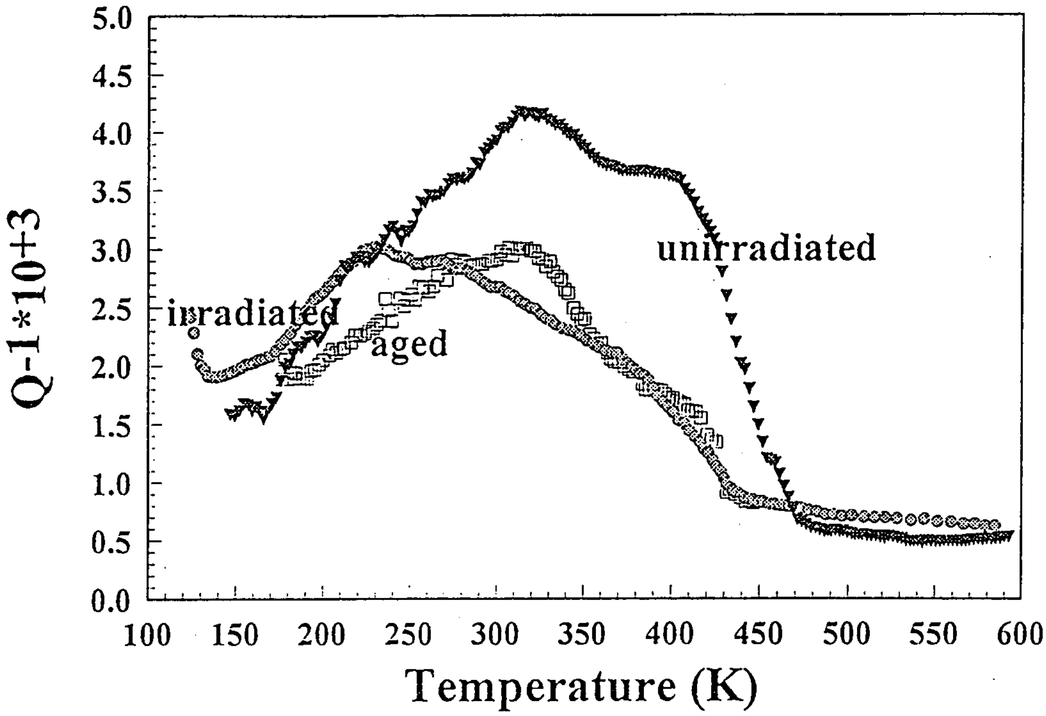

Figure 11. Effect of Ageing and Irradiation on Doel-I,-II Base Metal

Internal friction Q^{-1} can be considered proportional to Λl^2, where Λ is the mobile dislocation density and l is the dislocation segment length. At 150-175K, the dislocations have sufficient energy to overcome the potential barriers. With increasing temperature, the dislocations will travel greater distances: thus, l will increase and so will the IF-level Q^{-1}. At $\approx$ 320K, interstitial atoms can become mobile and pin the dislocations, restricting their motion and thereby decreasing l and Q^{-1}. At even higher temperatures, small carbides may form and also pin the dislocations.

Irradiation reduces the Q^{-1} peak height and lowers the peak temperature. Irradiation is known to induce, among others, precipitation of substitutional atoms, which can pin dislocations, resulting in a lower peak height. Most remarkable on Figure 11 is the reduction of peak height after thermal ageing: it is similar in magnitude to the reduction induced by irradiation.
The processes responsible for the peak shift towards lower temperatures for the irradiated base metal - in contrast with the absence of such effect in the aged condition- remain to be thoroughly investigated. Clearly, these results deserve more elaborate research.

7.0 CONCLUSIONS.

In conclusion, it is anticipated that this R&D effort, combining the investigation of trepans from two decommissioned vessels and of relevant specimens from accelerated irradiations, will provide a <u>*comprehensive demonstration*</u> of the proposed ***"Enhanced Surveillance Strategy"***, including the benefit of complementing current power plant surveillance by selected BR2 exposures, in order to define ***physically-grounded, plant-specific fracture toughness trend curves.***

It is contended that systematic implementation of "**Enhanced Surveillance**" will allow:

- **Significant Decrease of any Unwarranted Regulatory conservatism**
- **Major cost savings** (e.g.: heat-up and cooldown limitations)
- **Better plant life management decisions,** associated to **more adequate safety margins.**

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