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The Role of Cu in Radiation Embrittlement of LWR Pressure Vessel Steels

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경수로 압력용기 조사취화에서의 Cu의 역할

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1. Introduction

It is well known that exposure to high energy neutrons in the beltline region of a reactor can lead to significant mechanical property changes in the RPV (reactor pressure vessel) steels, most notably, degradation of fracture resistance or embrittlement.

This embrittlement phenomenon increases the probability of rapidly propagating fracture and, in the end, affects the life span of a reactor.

In this respect it is important to understand irradiation embrittlement mechanism and to develop a model for embrittlement based on detail microstructural evolutions in irradiated RPV steels for predicting the trend of mechanical property changes and in planning proper countermeasures for the safe operation of a reactor during life-time.

However, even though innumerable experimental and theoretical studies have been performed on irradiation embrittlement of RPV steels, because of the potentially serious nature of embrittlement observed, the early studies were largely concentrated on engineering implicatons in order to minimize the negative consequences of such effects in nuclear power plants. In addition, partly because of the complexity of factors contributing to embrittlement and partly of the inability to identify the defects responsible for irradiation hardening directly by TEM (transmission electron microscope), the damage microstructure has not been well characterized. Due to these reasons, the embrittlement phenomenon has been recognized as "well known but not well understood phenomenon"[1].

In particular, in relation to the role of Cu element in irradiation embrittlement of RPV steels, its detrimental effects have been well recognized as a major contributor to embrittlement for about 20 years, but detail behavior of Cu in RPV steels are still not well characterized.

Recently, based on a significant data base on radiation hardening and with the help of powerful research tools such as FIM/AP (Atom prove field ion microscope), SANS (Small angle neutron scattering) and TEM, some ambiguous nature of residual elements and defects in irradiated RPV steels has become clarified and theoretical modelling of embrittlement based on irradiated micros-

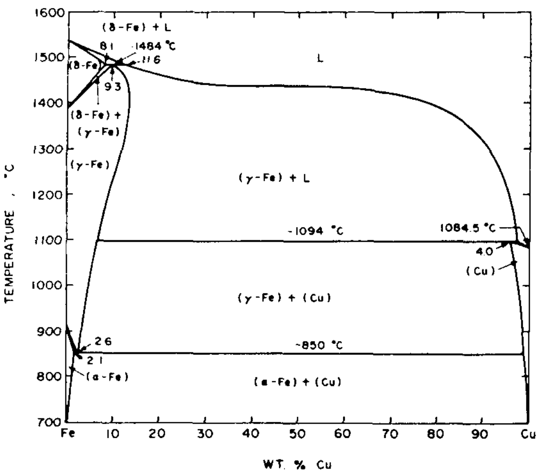

Fig. 1. The Fe-Cu Phase Diagram [2].

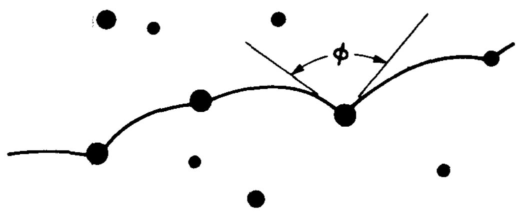

Fig. 2. Dislocation Held up at Dispersed Particles or Precipitates, Showing Definition of the Bowing Angle ϕ [2].

tructural characteristics has become possible.

Since understanding the cause of embrittlement, as well as predicting its severity, is essential for estimating the interity of RPV and, in addition, knowledges on irradiation embrittlement of RPV steels are required for various fields such as reactor design, reactor safety analysis, PWR life extention program and regulatory work, the authors intended to introduce the over-all embrittlement mechanisms and two well known models for RPV embrittlement, focusing on the role of Cu in embrittlement. In addition, we compared the embrittlement behavior of Kori unit-1 weld metal with model prediction. The authors wish this technical paper be helpful to those who are interested in irradiation embrittlement of RPV steels, especially of high Cu weld metal of Kori unit-1.

2. Hardening due to Cu under unirradiated condition

Understanding the role of Cu in Cu-containing steels under unirradiated condition and its contribution to matrix hardening is important to understand the irradiation embrittlement phenomenon.

Fig. 1 shows the Fe-Cu phase diagram. On cooling, the solubility of Cu in α-Fe decreases rapidly with decrease in temperature, providing the opportunity for subsequent precipitation on age hardening to be employed.

Of the strengthening mechanisms, the particular concern in connection with Cu-containing steels are solid solution and precipitation hardening.

It appears that Cu raises the yield point of ferrite to the extent of 3.8 Pa for each 0.1 % Cu present and the total effect is relatively small owing to the limited solubility in solid solution hardening.

As seen in Fig. 1, Cu solubility decreases less than 0.03 wt % at 300 °C. As temperature decreases coherent BCC Cu-rich clusters are formed and transformed to FCC ϵ-phase particle. It is this ϵ-phase particle that contribute to the precipitation hardening of Cu-containing alloys.

In Fe-Cu alloys it is revealed that Cu precipitation can increase the yield strength of ferrite up to 248 MPa per 1 % Cu compared to 3.8 Pa for each 0.1 % Cu in solid solution [2].

2.1. Russell-Brown Model [3]

At present, proposed two principal models for RPV irradiation embrittlement explain the contribution of Cu element to hardening through the Russell-Brown model. The model suggests that strengthening in Fe-Cu alloys is due to the difference in elastic moduli of particle and matrix (modulus hardening). As in Fig. 2, when a dislocation is cutting a spherical precipitate with an elastic modulus lower than the matrix, they showed that the strength of the material is given by

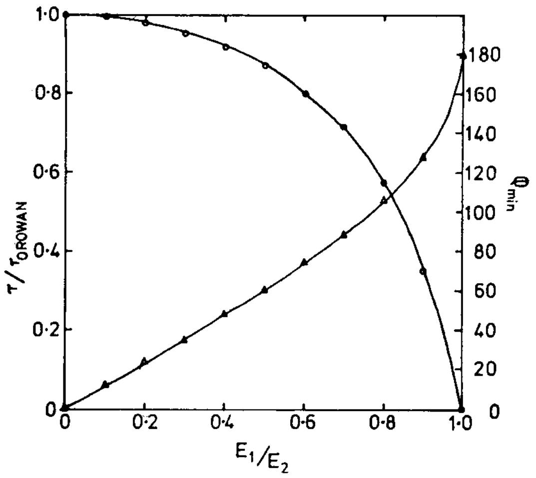

Fig. 3. Strength (Circles) and Cutting Angle (Triangles) as a Function of E_1/E_2. When $E_1/E_2=0$ the Precipitate is a Void, Which is a Strong Obstacle and Results in the Orowan Strength. When $E_1/E_2=1$, no Strengthening Occurs [3].

$$\tau = \frac{G \cdot b}{L}\left[1-\frac{E_1^2}{E_2^2}\right],\ \mathrm{Sin}^{-1}\frac{E_1}{E_2} > 50^\circ$$

where τ = Shear stress

G = Shear modulus

b = Burgers vector

L = Particle spacing

E_1, E_2 = Dislocation energy in particle and matrix, respectively. ($E \sim Gb^2$)

Fig. 3, shows the strength and cutting angle as a function of E_1/E_2. It is seen that precipitation hardening system with value of E_1/E_2 equal to 1/2* or so can cause a strengthening of only 10 % below the full Orowan strengthening. L.M. Brown and R.K. Han [4] calculated the strength increment ($\Delta\tau$) when the precipitates are cut by dislocation and suggested that $\Delta\tau$ may be given as

$$\Delta\tau = \left(\frac{1.6G \cdot b}{\ell}\right)\left(\frac{f}{\pi}\right)^{1/2}\left[\cos\left(\frac{\phi}{2}\right)\right],\ \phi < 100^\circ$$

$$\Delta\tau = \left(\frac{2G \cdot b}{\ell}\right)\left(\frac{f}{\pi}\right)^{1/2}\left[\cos\left(\frac{\phi}{2}\right)\right]^{3/2},\ \phi > 100^\circ$$

* shear modulus ratio of $\frac{Cu}{Fe} = \frac{4.85\times10^4[\mathrm{MNm^{-2}}]}{8.30\times10^4[\mathrm{MNm^{-2}}]} \doteqdot 0.58$

where, G is the shear modulus, b the burgers vector of the matrix, ℓ the particle diameter, f the volume fraction of particle and ϕ the bowing angle of cutting dislocation, as shown in Fig 2. The value of ϕ, when cutting takes places, is generally assumed to be constant in particular alloys and it has been demonstrated that ϕ depends on the ratio of the shear moduli of precipitate and matrix [5].

This findings led Russel and Brown to the adoption of the concept of modulus hardening when they proposed a theory which is able to account for both the observed yield strength and the work hardening behavior in Fe-Cu alloy system.
They found that maximun strength is given by

$$\tau\, \max = 0.041\ G\ b\, f^{1/2}/r_o$$

where, r_o is core radius of dislocation.

They demonstrated through experiments on Fe-Cu alloys that the maximun strength which can be achieved is proportional to the square root of the volume fraction of precipitate. Fig. 4 and Fig. 5 show excellent agreement between theory and experiments.

From Russel-Brown theory, a conclusion can be drawn as follow: In Fe-Cu alloy, the strength is achieved solely by particle cutting and the strength is proportional to the square root of the volume fraction of precipitate and the difference in elastic modulus of Cu precipitate and Fe matrix.

3. Microstructural Evolution under Irradiation Condition and The Role of Cu Element

At present the manner in which alloying/impurity elements contribute to the microstructural changes responsible for embrittlement remains unclear. One of the reason is that TEM examination

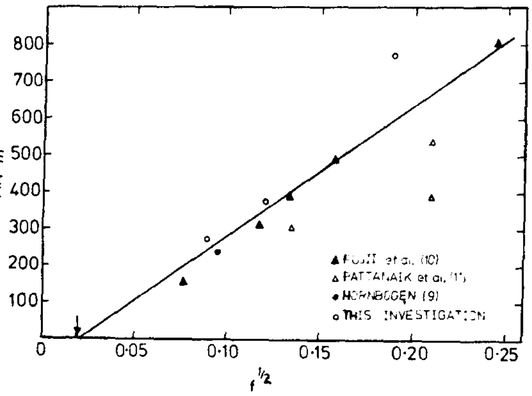

Fig. 4. Plot of Maximum Increase in Yield Strength vs. $f^{1/2}$ for Aging Temperatures 475-525°C. The Line Drawn is Predieted by the Theory if the Dislocation Core Radius is Taken Equal to 2.5 [3].

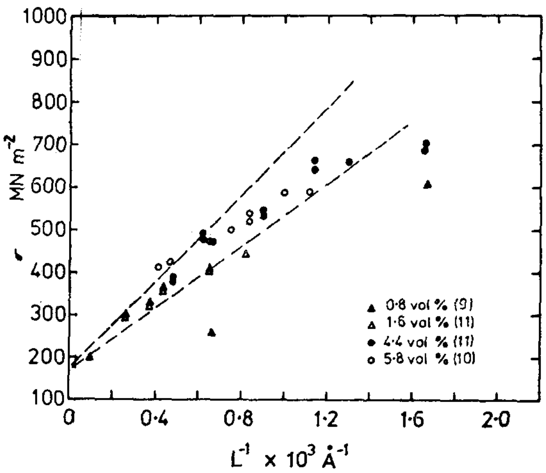

Fig. 5. Plot of Yield Stress vs. L^{-1} From Published Data. The Lines Drawn are Upper and Lower Limits Predicted by Theory [3].

of embrittled steels have failed to reveal the neutron-induced defects [6]. This means that their size is either below that of the resolution limit of the technique ($\sim 1 \sim 1.5$ nm dia. in good ferrite foils) or they induce insufficient strain or absorption contrast to be imaged [7]. However, other techniques have been more successful. For example, radiation induced volumes $<$ 10 nm dia. enriched in Cu or P have been detected in pressure vessel steels by FIM/AP [8], and the presence of small radiation-induced defects $<$ 6 nm dia. in pressure vessel steels and Fe-Cu model alloys has been deduced from SANS experiment.

In SANS experiment, various interpretations of the scattering have been made in terms of microvoid or dislocation loop formation, the radiation-enhanced precipitation of Cu or CuFe clusters, or the clustering/precipitation of P in association with point defects or as phosphides [9-13].

Of these candidate defects or clusters which are supposed to contribute to embrittlement of RPV steels, most experimental evidences are given to

1) Cu and P precipitate,
2) Vancancy clusters/Microvoid,
3) Defect complexes and interactions.

Following is a brief summary of recent findings on the nature of defects which are identified as contributors to embrittlement [14].

— Cu precipitates

Along with the role of Cu in unirradiated Fe-Cu alloys, Cu precipitates are regarded as dominant contributors in the irradiation hardening of RPV steels containing significant concentrations of this impurity.

As seen in Fig. 1, Fe-Cu alloy has very low solubility at 300°C, i.e$<$0.03 wt %. With this low solubility, very large acceleration of diffusion of Cu under irradiation due to vacancies lead to rapid precipitation of Cu in the early stage of irradiation.

The acceleration of diffusion of Cu in matrix under irradiation can be estimated by comparing the diffusion coefficient under irradiation (D^{irr}_{sol}) and unirradiation(D^{th}_{sol}) [15].

$$\frac{D^{irr}_{sol}}{D^{th}_{sol}} = \frac{2\eta G_{dpa}}{S_t \cdot D_{sd}} + 1$$

where

η Gdpa = interstitial, vacancy production rate by irradiation

S_t = total sink strength

D_{sd} = self-diffusion coefficient

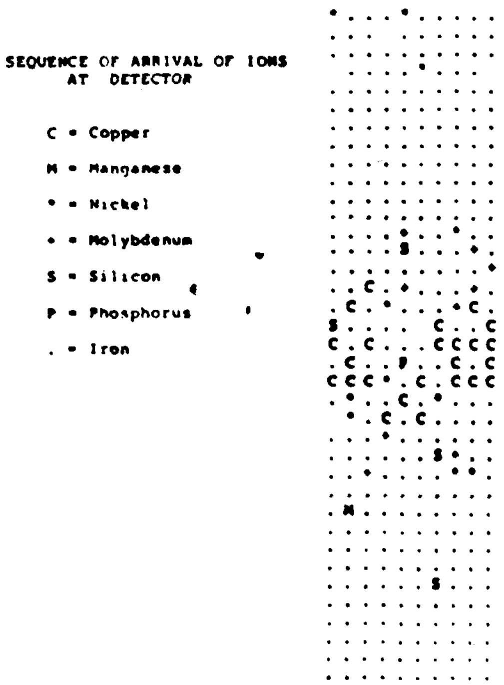

Fig. 6. Cluster Formed in HBR2 Weld. Note Concentration of Cu, Ni, and Mn [24].

With proper values for the above equation it was found that very large acceleration of diffusion can occur under irradiation, e.g. $D^{irr}_{sol}/D^{th}_{sol}=8250$ [15].

Odette [15], Fisher [16] and *et al* [10,17,18] identified radiation enhanced diffusion accelerated precipitation of Cu-rich phase as dominant mechanism of embrittlement. (later in section 4, Cu-ppts contribution to hardening appears as $\Delta \sigma_{Cu}$ in the Fisher Model for RPV embrittlement)

Fairly convincing evidences of irradiation induced precipitates formation in model alloys were revealed from recent SANS studies [10,17,19,20], TEM investigations of model alloys either containing high levels of Cu (>0.6 wt %) [10] or which have been thermally aged to coarsen the precipitate structure in low Cu alloys [21] and FIM measurements [22].

But the character of the defects in steels is still more ambiguous [10,20,23].

— Cu clusters

S.P Grant, S.L. Earp *et al* [24] identified Cu clusters highly enriched with Ni and Mn by Atom Prove Microchemical Analysis in two irradiated low alloy steel welds. Fig. 6 shows clusters formed in weld.

— Vacancy clusters/Microvoid

A variety of TEM [10,24], SANS [10,12], FIM/AP [22] and Positron Annihilation (PA) [25,26] studies have indicated the presence of small vacancy clusters in irradiated model Fe-alloys. Available SANS [10,12,20] and some PA data [27] on commercial steels also are consistent with a vacancy component of damage. Thermal annealing studies also suggest vacancy component of damage since vacancy recover prior to significant thermally induced precipitate coarsening.

In relation to microvoid, it is believed that a fraction of vacancy clusters (50-400 vacancies) would grow as microvoid. This explains the apparent increase in hardening and shift in low-Cu steels [14].

— Defect complexes and Interactions

Odette and Lucas [14] suggested the possibility of forming vacancy-solute complexes due to intracascade clustering and segregation. They explained that long-range migration of defects and solutes to sink (Radiation-Induced Segregation) could result in the formation of complexes and/or alloyed precipitates.

They also discussed that coupled with a significant time-at-temperature contribution, cluster sinks would contribute to a shift in precipitation to higher fluences with increasing flux.

Eventhough the effect of flux on microvoid formation is more complex and can not be simply estimated, currently it is expected that lower flux might be expected to reduce microvoid formation [12].

At the moment there is no clear understanding especially regarding to interactions between the various defect species. Odette [14] discussed that some clue may be obtained through a post-irradiation annealing experiment (PIA) because recovery would proceed at both increasing time and temperature in the sequence of clustering annealing, microvoid annealing and precipitate coarsening and dissolution, with the amount of Cu resolution depending on the annealing temperature.

However, as excess vacancies from annealing of clusters and small microvoid would enhance precipitate growth or coarsening and may induce the growth of larger microvoids, even more complex interactions might be postulated, such as excess vacancy enhanced Cu diffusion to dislocation, possibly coupled with dislocation climb, followed by pipe diffusion-dominated precipitate growth and coarsening kinetics.

Because of its complexity in alloying elements and structure few experiments have been made for irradiated RPV steels compared with model alloys. So it is not difficult to imagine that much more complex defect structure will be evolved in irradiated RPV steels. Thus, we can't rule out the possibility that all of defects mentioned in this section will be evolved in irradiated RPV steels.

In conclusion, detailed understanding and modelling of microstructural evolution will require better characterization of the microstructure and a detailed exploration of the factors which control their evolution.

4. Modelling for RPV Irradiation Embrittlement

One of the ultimate purpose of the study on damaged microstructures such as reviewed in Section. 3 is to develop a model which can predict the future trend of mechanical property changes through the understanding on the microstructural features induced by irradiation and the relationship between these microstructural features and macro-mechanical property changes.

Two principal models have been proposed for the prediction of embrittlement effects in pressure vessel steels, viz. that of Odette and coworkers [18,28] and that of Fisher *et al* [29,30].

In both models allowance is made for a radiation damage component (independent of Cu content) and a component related to radiation enhanced precipitation of Cu for Cu contents >0.1 wt %.

The Odette model has been tested by fitting the data available in the EPRI (Electric Power Research Institute) irradiated pressure vessel steel data base [31] and has provided a sound basis for the calculative procedures for adjustment of reference temperature given in Revision 2 of Regulatory Guide 1.99 [34]. Later, Odette revised his original model which was developed from the statistical analysis of surveillance data [36]. In the revised model Odette intended to develope better embrittlement forcasting methods through a synthesis of various sources of information including: physical models; fundamental experiments; test and power reactor Charpy-V-notch and other mechanical property data; and statistical regression. Odette assumed in the revised model that the evolution of a damage micro-structure includes both copper precipitates (as important and dominant one) and a radiation damage component (i.e, small vacancy clusters in the form of microvoids, depleted zones, or dislocation loops) enhanced by nickel. In the revised model total shift in the Charpy-V-notch transition temperature at the 41 Joule energy level (ΔT) is given as the sum of impurity (Cu, Ni) precipitate contribution (ΔT_i) and a radiation damage contribution that is independent of copper and that is enhanced by nickel (ΔT_d) as follow:

$$\Delta T = \Delta T_i(\mathrm{Cu},\ \mathrm{Ni},\ T_{irr},\ \phi,\ \phi t) + \Delta T_d(\mathrm{Ni},\ \phi t,\ T_{irr})$$

ϕ = neutron flux

ϕt = neutron fluence

T_{irr} = irradiation temperature

Detailed description of the model can be found in

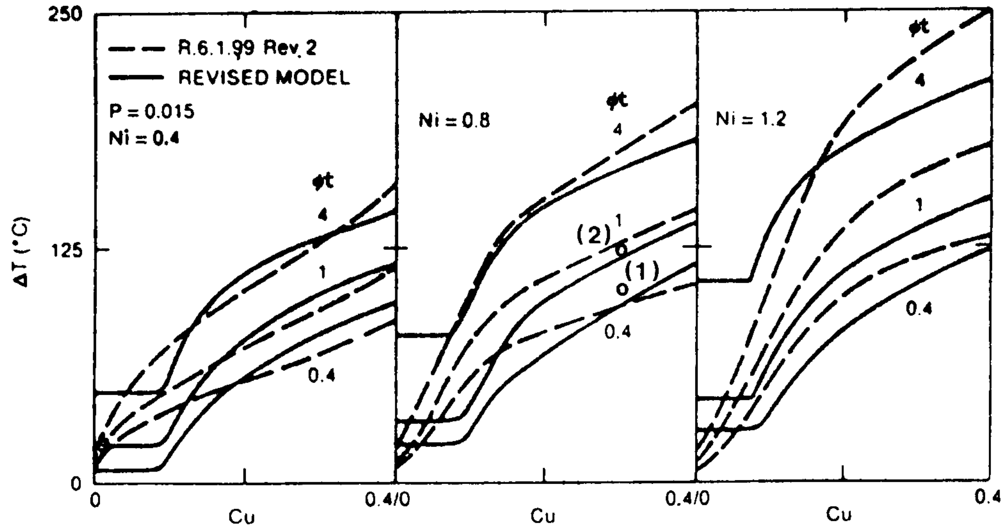

Fig. 7. A Comparison of the Revised Model Predictions and the R.G. 1.99R2 Mean Shift Value [33].

references 33 and 36. Fig. 7 shows a comparison of the revised Odette model predictions and the Regulatory Guide 1.99 Rev. 2 mean shift value. T(°C) is the ductile-brittle transition temperature shift in Charpy-V-notch energy indexed at 41 Joule and ϕt is the neutron dose expressed in 10^{23} n/m^2. Except at low Cu level, the comparison give good result within 10°C at all nickel levels.

The Fisher model was developed for the interpretation of surveillance data from the U.K Magnox monitoring scheme. The nature of the radiation damage component is not specified, but its effect is predicted, by reference to the measured dose and temperature dependence of hardening in mild steels after accelerated irradiation, using the relationship, $\Delta\sigma_y = A_i\sqrt{\phi t}$, where ϕt is the neutron dose and A_i is a composition and temperature dependent multiplier.

The Cu contribution is predicted from the Russel and Brown theory [3] after taking account of an appropriate acceleration of precipitation produced by the radiation-induced increase in equilibrium vacancy concentraton. Fisher *et al* [16] *suggested* that the radiation enhanced vacancy concentration is dependent on neutron dose rate, temperature, the yield of mobile vacancies per damage cascade, vacancy diffusivity and the point defect sink density (related to dislocation density, interface area, etc).

The Fisher model describes the total change in yield stress at time t ($\Delta\sigma_{tot}$(t)), in irradiated steels containing Cu, as follow.

$$\Delta\sigma_{tot}(t) = \Delta\sigma_{dam}(t) + \Delta\sigma Cu(t)$$

$$\Delta\sigma_{dam}(t) = A[\alpha_{b\cdot s}(\phi_f \cdot t)^{1/2}]$$

$\Delta\sigma_{Cu}(t)$ = (i) for $t < 0.05\ t_p$:

$$\Delta\sigma_{Cu} = 0$$

(ii) for $0.05\ t_p < t < t_p$: $\Delta\sigma_{Cu} = 0.77\ \log(t/0.05t_p)\cdot\Delta\sigma_{Cu}^{max}$

(iii) for $t > t_p$:

$$\Delta\sigma_{Cu} = -0.05\ \log(0.01\ t/t_p)\cdot\Delta\sigma_{Cu}^{max}$$

$$\Delta\sigma_{Cu}^{max} = 4.5\times 10^3\ f^{1/2} - 90\ (\mathrm{MN\ m^{-2}})$$

$\Delta\sigma_{dam}(t)$ = contribution from damage clusters

$\Delta\sigma_{Cu}(t)$ = contribution from Cu ppts

$\Delta\sigma_{Cu}^{max}$ = peak copper precipitate contribution

A = parameter related to material (steel composition) and irradiation temperature

f = volume fraction of Cu in steel

t = irradiation time

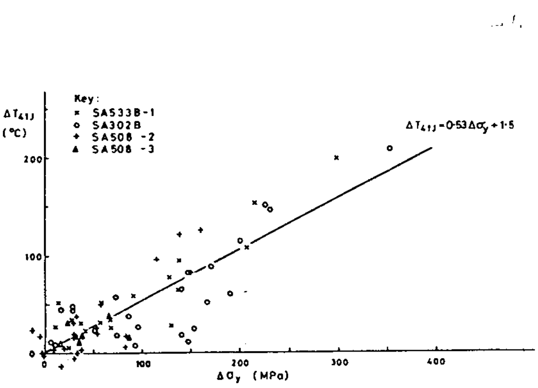

Fig. 8. Transition Shift (ΔT_{41J}) as a Function of the Change in Yield Stress, $\Delta \sigma_y$. Surveillance Irradiation at ~290°C. Tensile Testing at Room Temperature [16].

t_p =time for the precipitate dispersion to reach peak strength

$\alpha_{b \cdot s}$ =constants that reflect the difference in the efficiency of damage loop production between different spectra referring to below and side core locations.

ϕ_f =equivalent fission flux

For more detailed structure of the model, see reference 16.

Originally the Fisher model was developed to allow an interpretation of yield stress change ($\Delta \sigma_y$) under irradiation. But many of the data from PWR surveillance or MTR (Material Testing Reactor) tests are in the form of measured shifts in transition temperature (ΔT). The relationship between $\Delta \sigma_y$ and ΔT may be found from a consideration of the theory of brittle fracture developed by Cottrel [35]. Fisher determined the relationship between $\Delta \sigma_y$ and ΔT using the data from PWR surveillance irradiations at 290°C. Fig. 8 shows the result, i.e., $\Delta T_{41J}=0.53\ \Delta \sigma y+1.5$, where ΔT_{41J} is again Charpy-V-notch transition temperature indexed at 41 Joule.

Figs 9, 10 and 11 show good examples that can

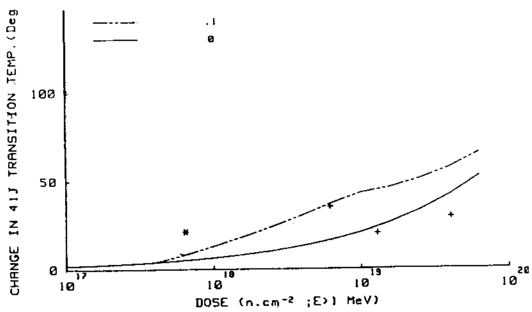

Fig. 9. Predicted ΔT_{41J} as a Function of Dose Compared with Additional Surveillance Data from Steels with Copper Contents 0-0.1 wt%. Data Associated with Nickel Contents >0.64 wt% are Asterisked [16].

examine both the predictablility of the Fisher model and the deleterious influence of copper and nickel.

It can be seen that the model successfully reproduces the trends of the data with increasing exposure and nickel shows a deleterious influence for intermediate and high copper. Especially Fig. 11 shows clearly the deleterious effect of Cu. Data used for these figures can be found in reference 16.

Finally, a short comment will be made on the embrittlement behavior of Kori unit-1 weld to help reader's understanding on the integrity of domestic RPV steels. In Figs 7 and 11, numbered open circles indicate data on weld metal of RPV surveillance capsules. They were incorporated to compare and to evaluate the embrittlement of Kori unit-1 weld (Cu=0.29%, Ni=0.68%). No. 1 tested at 1.1 EFPY (effective full power year) and No. 2 at 5.1 EFPY, respectively. Data show explicitly that the embrittlement of Kori unit-1 weld metal reside within the scope of model prediction. In other words, it can be seen that the two models adequately describe the trend of embrittlement of Kori unit-1 weld. Detail descriptions are beyond the scope of this report. Those who are interested in the embrittlement behavior of domestic RPV

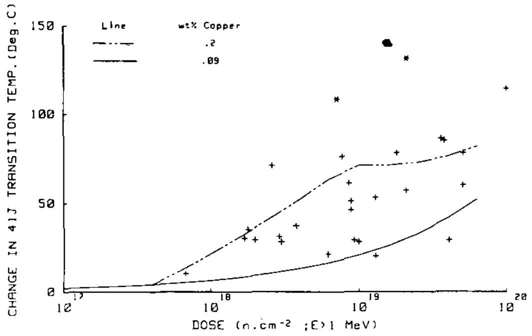

Fig. 10. Predicted ΔT_{41J} as a Function of Dose Compared with Additional Surveillance Data from Steels with Copper Contents 0.09-0.2 wt%. Data Associated with Nickel Contents >0.64 wt% are Asterisked [16].

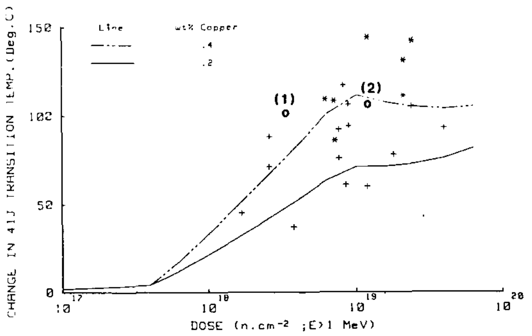

Fig. 11. Predicted ΔT_{41J} as a Function of Dose Compared with Additional Surveillance Data from Steels with Copper Contents 0.2-0.4 wt%. Data Associated with Nickel Contents >0.64 wt% are Asterisked [16].

steels are encouraged to see reports on RPV surveillance test.

5. Conclusions

Knowledge of the features underlying the embrittlement in RPV steels during irradiation has grown markedly in the past few years. In addition, embrittlement forcasting capability also has grown markedly with those knowledges.

Current understanding from indirct evidence and a combination of preliminary and individually imperfect or incomplete observations tell us that hardening microstructures are predominantly characterized by radiation enhanced diffusion accelerated Cu (or Cu-rich) precipitates and microvoids or a complex of these and other unidentified constituents. These views contrast to the early understandings on the behavior of Cu element in that the latter referred to a stable copper-vacancy defect [32].

A sound physical understanding of embrittlement requires a quantification of mechanisms which relate changes in basic structure sensitive properties to Charpy V-Notch shifts (or other fracture related properties), microstructural changes to basic properties, and irradiation and metallurgical evoluton. Of these, it seems that rigorous characterization of the radiation induced microstructure, a basic prerequisite for resolving the latter two problems, has yet to be achieved. So, it is believed that a better characterization of the microstructure and a detailed exploration of the factors which control their evolution is needed for the clear understanding of RPV embrittlement mechanism. L.M. Davies and T. Ingham identified additional features that need to be investigated further [37]. It includes, for examples, the role of Ni, Mo_2C precipitates, phosphorus in addition to Cu.

Finally, a short comment will be made on current domestic research state in this field. Since Kori unit-1, several RPV surveillance tests have been performed for operating reactors according to the requirement of domestic Atomic Energy Law and contingent elementary studies also have been made by KAERI (Korea Advanced Energy Research Institute). More basic research on RPV irradiation embrittlement, however, has not been initiated due to various reasons. One of these reasons is the absence of a high flux research reactor. With the start of multipurpose research reactor (MRR) in 1993, however, it is expected that active research will be made on radiation effects on various nuclear materials as well as on RPV radiation embrittlement.

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