Correlation among the Changes in Mechanical Properties due to Neutron Irradiation for Pressure Vessel Steels

Kunio ONIZAWA and Masahide SUZUKI

Department of Reactor Safety Research, Tokai Research Establishment, Japan Atomic Energy Research Institute, Tokai-mura, Ibaraki-ken, 319-11 Japan.

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Irradiation hardening and embrittlement of reactor pressure vessel (RPV) steels having different contents of copper and nickel have been investigated as a part of the phase III of IAEA Coordinated Research Program (CRP). Seven kinds of materials which were made by Japanese steel manufacturers for the CRP were used. Neutron irradiation was conducted in Japan Materials Testing Reactor (JMTR). Mechanical properties of the materials were examined by conducting the hardness, tensile, Charpy impact and fracture toughness tests before and after irradiation. Some relationships between the changes of the mechanical properties due to irradiation were established. The increase in yield strength was correlated with hardness increase and the shift of Charpy transition temperature. In the upper shelf temperature range, the decrease in fracture toughness was well correlated with the degree of the increase in yield strength.

KEY WORDS: pressure vessel steel; neutron; irradiation embrittlement; chemical composition; mechanical property; transition temperature; fracture toughness; Charpy impact test; structural integrity; surveillance.

1. Introduction

The life time of nuclear power plant is greatly dependent on the operational life of major components that are difficult to repair and replace. The reactor pressure vessel (RPV) is one of the important components that the integrity should be maintained at highest degree to continue safe operation. To assess the integrity of an RPV, material degradation due to aging, in particular, neutron irradiation embrittlement of the steel should be evaluated quantitatively and precisely. In this respect, the prediction and the evaluation of mechanical properties, such as Charpy impact property and fracture toughness of the steels are very important.

Extensive studies on irradiation embrittlement of RPV steels have been performed since nuclear reactor was commenced to operate. The IAEA Coordinated Research Program (CRP) on irradiation embrittlement of pressure vessel steels was initiated in 1971, and the phase II and the phase III of CRP were continued afterwards.^{1–3)} The phase III of CRP, entitled “Optimizing of Reactor Pressure Vessel Surveillance Programmes and their Analysis,” was initiated in 1983. Fifteen countries participated in the phase III program. The Japan Atomic Energy Research Institute (JAERI) took part in the program and performed the experiments on irradiation embrittlement of RPV steels. The results of the experiments performed at JAERI have been described elsewhere.⁴⁾

The mechanical tests of irradiated specimens are usually limited to a small number of the iteration. To determine the proper testing conditions, it is desirable to

predict the mechanical property before the test by means of correlation between the other property. The correlation also helps to obtain more reliable results. Several predictive equations for the shift of Charpy transition temperature have been developed based on the analyses of surveillance test results, *e.g.* Ref. 5). These equations were formulated based primarily on chemical composition of steels and fast neutron fluence values. However, the prediction formula for fracture toughness change has not yet been established. On the other hand, the microstructural studies on radiation damage of RPV steels have been performed mainly for the irradiation hardening, *e.g.* Ref. 6). Therefore, the correlation between the irradiation hardening and the changes of different mechanical properties, in particular, fracture toughness should be established.

The chemical composition of RPV steels has changed with the improvement of the technology for manufacturing steels. The impurity elements in steel have been decreased gradually since the first generation of nuclear power plants was constructed. Hence RPV steels of the nuclear power plants which are now being operated have some variations in the level of impurity elements. The objective of this study is to investigate the correlation between different mechanical properties of irradiated materials having a certain range of alloying and impurity elements that cover the range of chemical composition for the Japanese PWR pressure vessel steels.

2. Experimental Procedure

2.1. Materials

Chemical compositions of the steels used are shown in **Table 1**. These materials designated as JPD, JPF, JPH, JPI, JPJ, JFL and JRQ were made by Japanese steel manufacturers for the phase III of IAEA CRP.⁷⁾ All materials were made as ASTM A533B class 1 plates, except for the JFL which was ASTM A508 class 3 forging. The materials have a wide range of chemical composition on the elements, copper, nickel and phosphorus, which are very important from the viewpoint of irradiation embrittlement of RPV steels. **Figures 1(a) and 1(b)** shows the compositional range of materials used in this

Table 1. Chemical composition of materials used in the present study. (wt%)

Element Material	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	Al	Spec.	Thick- ness (mm)
JPD	0.19	0.27	1.45	0.006	0.001	0.16	0.10	0.15	0.55	0.01	0.019	A533B1-Ni	30
JPF	0.18	0.27	1.49	0.020	0.001	0.16	0.62	0.15	0.55	0.01	0.010	A533B1	30
JPH	0.18	0.27	1.45	0.006	0.001	0.17	1.18	0.15	0.54	0.01	0.021	A533B1+Ni	30
JPI	0.18	0.21	1.50	0.006	0.001	0.01	0.69	0.17	0.57	0.001	0.012	A533B1	163
JPJ	0.18	0.21	1.41	0.005	0.001	0.05	0.64	0.10	0.50	<0.003	-	A533B1	250
JFL	0.17	0.25	1.44	0.004	0.002	0.01	0.75	0.20	0.51	0.004	0.016	A508-3	290
JRQ	0.18	0.24	1.42	0.017	0.004	0.14	0.84	0.12	0.51	0.002	0.014	A533B1	225

Fig. 1. Copper vs. nickel (a) and phosphorus vs. copper (b) contents in steels used in the present study. Estimated ranges of the Japanese PWR steels are also drawn as hatched regions.

study together with that of Japanese PWR pressure vessel steels. Three kinds of laboratory melted alloys (JPD, JPF and JPH) were manufactured in 30 mm thickness. The other heavy section steels were with thickness from 163 to 290 mm. Heat treatments of these steels are summarized in **Table 2**. For the heavy section steels (JPI, JPJ, JFL and JRQ), the specimens for the mechanical tests were machined from the quarter thickness position of materials. The cooling rate at quenching for the laboratory melted materials was controlled to be almost same as the rate at the quarter thickness position of the heavy section steels. Post weld heat treatment (T2) was applied to the heavy section steels. Microstructures of materials are almost identical, *i.e.*, bainitic structure with a small amount of ferrite.

2.2. Neutron Irradiation

Test specimens from materials mentioned above were irradiated in the Japan Materials Testing Reactor (JMTR, thermal output 50 MW). The irradiation was performed by using eight capsules that contained electric heaters, thermocouples and fluence monitors. All capsules were irradiated at the same irradiation hole in the second Beryllium reflector region of JMTR core. The average values of neutron fluence in the seven capsules were $\sim 2 \times 10^{19}$ n/cm² ($E > 1$ MeV), whereas the fluence for one capsule was $\sim 4 \times 10^{19}$ n/cm² ($E > 1$ MeV). The fluence value at each specimen position was evaluated by the values obtained from four fluence monitors in the capsule and the database of the fluence values determined by past experiments at the same irradiation hole. The ratio of neutron fluence with energy greater than 0.1 MeV to that for 1 MeV was approximately 2.6. Displacement per atom (dpa) for iron by the irradiation to 2×10^{19} n/cm² ($E > 1$ MeV) was about 0.031. The fast neutron flux was approximately 1×10^{13} n/cm²s ($E > 1$ MeV) for all capsules. The temperature of specimens during irradiation was controlled in the range from 280 to 300°C for all capsules.

2.3. Charpy Impact, Tensile and Hardness Tests

Charpy impact tests were performed using a test machine with the capacity of 300 J installed in a hot cell. The machine was designed according to JIS B7722 (Japan Industrial Standard B7722, similar to the related ISO standard). The configuration and dimensions of the specimen are shown in **Fig. 2(a)** which are also in

Table 2. Heat treatment condition of materials used in the present study.

Material	Heat Treatment Conditions		
JPD	N: 880 °C x 1 h AC, T1: 670 °C x 80 min AC		
JPF			
JPH			
JPI	Q: 880 °C x 4 h WC, T1: 670 °C x 7.5 h AC, T2: 620 °C x 40 h FC		
JPJ	Q: 880 °C x 7.25 h WC, T1: 655 °C x 8.7 h AC, T2: 617.5 °C x 41.4 h FC		
JFL	Q: 880 °C x 9 h WC, T1: 640 °C x 9 h AC, T2: 610-630 °C x 16.5 h FC		
JRQ	Q: 880 °C x 3 h WC, T1: 665 °C x 12 h AC, T2: 620 °C x 40 h FC		

N: Normalizing

Q: Quenching

T1: Tempering

T2: Simulated Post Weld Heat Treatment

AC: Air Cooling

WC: Water Cooling

FC: Furnace Cooling

Fig. 2. Configurations and dimensions of test specimens.

accordance with JIS Z2202. The notch orientation of the specimen is T-L direction according to ASTM E399.⁸⁾

Charpy transition curves were determined by fitting all data to the following hyperbolic tangent equation.⁹⁾

$$CVN = A + B \times \tanh\{(T - T_0)/C\} \quad \dots\dots\dots(1)$$

where CVN: Charpy absorbed energy,

T : Test temperature,

A, B, C, T_0 : Fitting constants.

Charpy upper shelf energy (USE) was determined as the average of all absorbed energy values that exhibited fully ductile tearing on the fracture surface.

Tensile tests were conducted using an electromechanical servo type test machine with the capacity of 100 kN. The tensile specimen has dimensions of 4 mm in diameter and 22 mm in gauge length (see Fig. 2(b)). The tensile direction of the specimen was perpendicular to the major working direction of materials. The yield strength of the materials was determined from the load at lower yield point. Vickers hardness tests were done using a half of broken Charpy specimen with an applied load of 98 N.

2.4. Fracture Toughness Tests

To measure fracture toughness, two types of specimen were used. One was a precracked Charpy-V type (PCCv) specimen (see Fig. 2(c)), and another was a compact type specimen (12.5 mm thick disk-shaped compact, 0.5T-DCT (Fig. 2(d)), and 25 mm thick standard compact, 1T-CT (Fig 2(e)). 1T-CT specimens were tested only in the unirradiated condition. Crack plane orientation of

all fracture toughness test specimens was T-L direction. Fracture toughness testing was performed based on ASTM E813 method.¹⁰⁾ Precracking into specimens was performed before irradiation using fatigue load of 10 Hz sinusoidal wave in air. Maximum stress intensity factor during precracking was kept less than $30 \text{ MPa m}^{0.5}$. The ratio of initial crack length to specimen width is 0.55 to 0.6. After precracking, side groove of 10% of thickness was machined in each side for compact type specimen.

In the ductile-brittle fracture transition temperature range, fracture toughness based on J-integral value was determined by the onset of cleavage fracture that corresponded to the sudden load drop. The fracture toughness (K_{Jc}) value was converted from the J-integral value at the cleavage fracture point.

The unloading compliance method according to ASTM E813 was applied to establish a J-integral resistance ($J-\Delta a$) curve using a single specimen method at upper shelf test temperature. The multiple specimen technique according to ASTM E813 was also used for making a $J-\Delta a$ curve in some conditions.

3. Results and Discussion

3.1. Increase in Yield Strength Caused by Neutron Irradiation

As is known well, Cu and Ni affect greatly the irradiation hardening and embrittlement. Increase in yield strength (σ_y) is caused by irradiation and is usually considered as a basic information on irradiation hardening. Figure 3 shows the increase in σ_y at room temperature

Fig. 3. Increase in yield strength caused by irradiation as a function of copper content in steels. [P], [Ni]: wt%, nvt: $\times 10^{19}$ n/cm², $E > 1$ MeV

Fig. 4. Increase in yield strength caused by irradiation as a function of nickel content in steels. [Cu], [P]: wt%, nvt: $\times 10^{19}$ n/cm², $E > 1$ MeV

caused by irradiation as a function of Cu content. It is clear that the increase in σ_y , that is, the irradiation hardening became larger with increasing Cu content, although there are some variations in nickel and phosphorus contents and neutron fluence (nvt) as indicated in the figure. For materials with high Cu content (0.16–0.17 wt% Cu), the σ_y increase tends to be affected also by Ni contents. This fact is more clearly seen in Fig. 4, where the σ_y increase is plotted as a function of Ni content. The irradiation hardening increases with increasing Ni content from 0.1 to 1.18 wt%. The trend of nickel dependence is consistent with the data in Ref. 11). Thus the irradiation hardening is used as a basic information for evaluating correlations between mechanical properties in this paper.

3.2. Relationship of Changes of Hardness and Charpy Impact Properties with Increase in Yield Strength

Figure 5 shows the correlation between the increases in σ_y and Vickers hardness (H_v) measured at room

Fig. 5. Correlation between the increases in yield strength and Vickers hardness by irradiation.

Fig. 6. Correlation between Charpy 41 J shift and yield strength increase by irradiation. The average α is 0.61 (°C/MPa).

temperature. The regression line is expressed by the following relation.

$$\Delta\sigma_y = 3.3 \times \Delta H_v \quad \text{.....(2)}$$

where $\Delta\sigma_y$: increase in yield strength by irradiation,
 ΔH_v : increase in Vickers hardness by irradiation
 correlation factor, $r = 0.84$.

This correlation means that the increase in σ_y caused by irradiation can be estimated from hardness values for materials used in this study. It can be applied to the RPV steels in the Japanese PWR.

For Charpy impact properties, a ductile-to-brittle transition temperature and an USE value are important as measures of irradiation embrittlement. Figure 6 shows the relationship between the shift of Charpy transition temperature at 41 J absorbed energy level, ΔT_{41J} , and σ_y increase at room temperature. The ΔT_{41J} of JPH was not obtained because of large scatter in the data around 41 J level. The relation is usually expressed by the linear equation as follows,

$$\Delta T_{41J} = \alpha \times \Delta \sigma_y \dots\dots\dots(3)$$

α : coefficient, the average value = 0.61 (°C/MPa)

The average value of α obtained in this study agrees well with Ref. 12), although α has a relatively large scatter from 0.4 to 0.9. Specimens from laboratory melts (JPD and JPF) and a steel with very low copper (JPI) that had low initial transition temperature below -70°C showed low α values. **Figure 7** shows the α value as a function of the initial Charpy transition temperature. It is seen that materials with low initial transition temperature show low α value, except for the JFL. This coincides with Refs. 12) and 13). The discrepancy in the JFL is not clear at this stage. Although influencing factors, *i.e.*, copper content in steel, initial transition temperature and manufacturing method, on this relation should be studied carefully, this type of equation helps to estimate the shift of transition temperature from tensile test results.

For an assessment of irradiation damage at the upper shelf temperature range, a decrease percentage of USE has been used in a prediction formula.⁵⁾ **Figure 8** shows the correlation between the decrease percentage of USE and σ_y increase. The USE values after irradiation was not determined for JPI, but the USE decrease was estimated to be less than 10% based on the load-time signals from instrumented Charpy impact tests. The relation between the USE decrease and σ_y increase is expressed by the following equation as is similar to the Eq. (3),

$$\Delta \text{USE} = \beta \times \Delta \sigma_y \dots\dots\dots(4)$$

β : coefficient, 0.17–0.55 (%/MPa) except for JPI

The range of β value, even except for the JPI, is broader than that of α value (0.4–0.9) as was shown in Fig. 6. The correlation between USE decrease and σ_y increase is somewhat weak when it is compared with the one between Charpy shift and σ_y increase, although both coefficients have some ranges.

Fig. 7. Relationship between the coefficient α ($=\Delta T_{41J}/\Delta \sigma_y$) and initial Charpy transition temperature. Low α values are seen for JPI with low Cu (0.01 wt%), and JPD and JPF which are the laboratory melted steels of 30 mm thick.

3.3. Fracture Toughness Change in the Ductile–Brittle Transition Temperature Range

Fracture toughness (K_{JC}) values in the ductile–brittle transition range were obtained before and after irradiation using PCCv and compact type specimens of JPI, JPI and JRQ. A statistical analysis is required to evaluate the K_{JC} transition curve, because there is a large scatter of K_{JC} value in the transition temperature range. Additional tests were performed for only JRQ to establish the K_{JC} curve for unirradiated material more precisely. **Figure 9** shows K_{JC} values from PCCv and compact specimens of JRQ before and after irradiation.¹⁴⁾ The mean K_{JC} curves were evaluated based on the master curve method including a statistical analysis,¹⁵⁾ assuming no change in the shape of K_{JC} curve after irradiation. The shift of the K_{JC} curve due to neutron irradiation was somewhat larger than that of the Charpy 41 J transition temperature. **Figure 10** shows the temperature dependence of the K_{JC} values obtained from PCCv specimens of JPI and JPI. The data bounding line for each condition

Fig. 8. Relationship between the decrease in USE and yield strength increase due to irradiation.

Fig. 9. Cleavage fracture toughness values of JRQ before and after irradiation. The mean curves for unirradiated and irradiated conditions based on Wallin's method¹⁵⁾ are also indicated.

Fig. 10. Cleavage fracture toughness values of JPI and JPJ obtained from precracked Charpy type specimen before and after irradiation. The data bounding line for each condition is indicated.

is also indicated. Precise shifts of K_{JC} curves could not be determined for these materials, because the number of test data was too small to perform a statistical analysis to determine the K_{JC} curves. The bounding lines, however, show that the shift of K_{JC} curve for JPJ is larger than that for JPI. It is also found that the shift of K_{JC} curve for JPJ is smaller than that for JRQ shown in Fig. 9. The increases in σ_y for these steels were the same tendency as the shift of K_{JC} curve. Therefore, the shift of K_{JC} curve is expected to be correlated with the increase in σ_y . In a code for integrity assessment of reactor pressure vessel, such as Section XI of ASME Boiler and Pressure Vessel code,¹⁶⁾ the shift of fracture toughness transition curve is assumed to be equivalent to that of Charpy impact energy curve. In this respect, it is necessary to verify the relation between the shifts of transition curves for fracture toughness and Charpy impact energy. The number of irradiated specimens for fracture toughness test is normally limited by the space for irradiation. It is desirable to optimize testing and evaluation methods for the determination of fracture toughness transition curve after irradiation from a small number of specimen. The investigation on the optimization of fracture toughness measurement is now underway.

3.4. Fracture Toughness Change in Upper Shelf Temperature Range

The J-integral resistance (J - Δa) curves of heavy section steels were evaluated at 100 and 200°C in the upper shelf temperature range based on the test results of two types of compact specimens. Figure 11 shows the relations of J-integral value and the amount of ductile crack extension, Δa , of JRQ at 200°C before and after irradiation. It is seen from the data for unirradiated specimens that there is no difference in the data between 0.5T-DCT and 1T-CT up to the ductile crack extension of ~0.5 mm. The amount of ductile crack extension almost corresponds to the length at which the J_Q is defined according to the ASTM standard. The crack

Fig. 11. Comparison of J-integral resistance curves of material JRQ at 200°C before and after irradiation.

Fig. 12. Comparison of J_Q values obtained from 0.5T-DCT and 1T-CT.

initiation toughness J_{IC} is obtained from the validation of the J_Q by some requirements like specimen size. Figure 12 compares the J_Q values for unirradiated materials obtained from 0.5T-DCT with those from 1T-CT. Valid J_{IC} was obtained at only limited condition of low fracture toughness from 1T-CT, because the requirement on specimen size was not fulfilled at high toughness level for such a small specimen. J_Q values of both 0.5T-DCT and 1T-CT including J_{IC} were, however, almost equivalent to each other, although the correlation factor r is not so high ($r=0.76$). On the other hand, the ductile crack growth resistance which corresponds to the slope of J-R curve, dJ/da , of 0.5T-DCT at the crack extension over 0.5 mm was slightly lower than that of 1T-CT as shown in Fig. 11. Therefore, the irradiation response on J_Q values is discussed below.

The J - Δa data of irradiated specimens were lower than those of unirradiated specimens as shown in Fig. 11. Figure 13 shows the J_Q values for each material as a function of fast neutron fluence. The average J_Q values

Fig. 13. Change in fracture toughness, J_Q , as a function of fast neutron fluence.

Fig. 14. Relationship between the decreasing slope of the regression line of fracture toughness shown in Fig. 13, $dJ_Q/d(nvt)$, and the increase in yield strength due to irradiation.

with a standard deviation before irradiation are also shown in the figure. Valid J_{IC} values were obtained only from JRQ steel, because other steels had very high toughness before and even after irradiation. The linear regression line for each material is also indicated in Fig. 13. Although the J_Q values after irradiation are scattered, it is found that neutron irradiation decreased the J_Q value. The decrease in J_Q value tends to be large for JFL, but lesser extent for JPI as clearly shown in the slope of the regression line.

In upper shelf temperature range, Charpy USE is used to evaluate the safety of reactor pressure vessel in some codes. For example, US 10 CFR part 50¹⁷⁾ stipulates that reactor vessel steel must maintain upper shelf energy of no less than 68 J throughout the life of the vessel. However, the integrity assessment of the vessel at the upper shelf temperature, *i.e.* around the operational temperature, requires practically the actual fracture toughness value, not Charpy absorbed energy value. Figure 14 shows the relation between the decreasing slope in fracture toughness, $dJ_Q/d(nvt)$, which

Fig. 15. Comparison of measured decrease of Charpy upper shelf energy and predicted one by the equation in the US NRC Regulatory Guide 1.99.⁵⁾

corresponds to the negative slope of the regression line in Fig. 13, and the increase in σ_y . This relation for three plate materials can be expressed by a dotted line as indicated in the figure. This correlation is better than the relationship between ΔUSE and $\Delta \sigma_y$, shown in Fig. 9. This suggests the advantage of predicting fracture toughness rather than Charpy USE from the σ_y increase for the integrity assessment of RPV. It is, however, necessary to make a further analysis on the effect of specimen size and the particular behavior of the JFL for the more reliable evaluation of the irradiation effect on fracture toughness.

From regulatory point of view, the decrease in USE should be evaluated as mentioned before. Figure 15 shows the relation between the predicted decrease in USE by the equation in the US NRC Regulatory Guide (R.G.) 1.99⁵⁾ and the measured one. This figure indicates that the R.G. 1.99 overestimates for JPI, JFL and JPD, which have either low Cu or low Ni contents. On the other hand, it underestimates for JPJ by 10%, which has also low content of Cu. Although the prediction equation in the R.G. 1.99 has been formulated with Cu content above 0.10 wt.% for base metal and neutron fluence, the decrease in USE may be varied with other variables. The correlation between the decrease in fracture toughness and irradiation hardening, therefore, is expected to be more useful for the integrity assessment of RPV than that between the decrease in USE and irradiation hardening.

4. Conclusions

As a part of the IAEA Coordinated Research Program, irradiation hardening and embrittlement of seven kinds of RPV steels containing different contents of Cu and Ni were investigated. Some mechanical property changes due to neutron irradiation were examined by conducting tensile, hardness, Charpy impact and fracture toughness tests. Thereby the responses of different mechanical properties were correlated. The increase in yield strength from tensile tests, which increases with

both Cu and Ni contents, was mainly used as a baseline of the relationship. The conclusions obtained are:

(1) The increase in yield strength can be correlated well with hardness increase and Charpy transition temperature shift.

(2) The shift of transition temperature in fracture toughness is somewhat larger than that in Charpy transition temperature.

(3) In the upper shelf temperature range, the decrease in fracture toughness is correlated well with the increase in yield strength.

(4) The decrease in upper shelf energy is not correlated well with the increase in yield strength.

It is noted that the optimization of the method to evaluate the fracture toughness from a small number of irradiated specimen is necessary.

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