



## The Evaluation of Ductile-Brittle Transition Temperature Before and after Neutron Irradiation in RPV Steels Using a Small Punch Test

Min-Chul Kim<sup>1)</sup>, Yong Jun Oh<sup>2)</sup>, Bong Sang Lee<sup>1)</sup>

<sup>1)</sup> Korea Atomic Energy Research Institute, Daejeon, Korea

<sup>2)</sup> Hanbat National University, Daejeon, Korea

### ABSTRACT

A small punch (SP) test was performed to evaluate the ductile-brittle transition temperature before and after neutron irradiation in reactor pressure vessel (RPV) steels produced by different manufacturing (refining) processes. The results were compared to the standard transition temperature shifts from the Charpy test and Master Curve fracture toughness test in accordance with the ASTM standard E1921. The samples were taken from 1/4t location of the vessel thickness and machined into a 10x10x0.5mm dimension. Irradiation of the samples was carried out in the research reactor at KAERI (HANARO) at about 290 °C of the different fluence levels respectively. SP tests were performed in the temperature range of RT to -196°C using a 2.4mm diameter ball. For the materials before and after irradiation, SP transition temperatures ( $T_{SP}$ ), which are determined at the middle of the upper and lower SP energies, showed a linear correlation with the Charpy index temperature,  $T_{41J}$ .  $T_{SP}$  from the irradiated samples was increased as the fluence level increased and was well within the deviation range of the unirradiated data. The  $T_{SP}$  had a correlation with the reference temperature ( $T_0$ ) from the master curve method using a pre-cracked Charpy V-notched (PCVN) specimen.

**KEY WORDS:** irradiation embrittlement, ductile-brittle transition temperature, small punch, reactor pressure vessel, SP transition temperature,

### INTRODUCTION

The transition temperature shift by irradiation in reactor pressure vessel (RPV) steels is of primary concern for reactor integrity. Charpy index temperature at 41J energy ( $T_{41J}$ ) has been used as a measure of the temperature shift by irradiation and indirectly positions the ASME  $K_{IC}$  curve on the temperature axis. Recently a direct method for determining the fracture toughness, named “Master Curve”, was developed and is receiving wide applicability to RPV steels [1-3]. In this method, the fracture toughness ( $K_{IC}$ ) with respect to the temperature is expressed uniquely as a single master curve shape and the temperature defined at  $K_{IC}=100\text{MPa}\sqrt{m}$  is used as a reference temperature for positioning the  $K_{IC}$  curve on the temperature axis. However, these standard methods require a significant volume of testing material. For the irradiated sample tests, specimen size is a critical concern because of the limited available surveillance material from the operating power plants and irradiated samples from the material test reactors. The limitation of specimen size also leads to difficulties in measuring the location-specific properties.

Small punch test might be a promising technique to overcome these limitations. Load-displacement curve from the small punch test involves several useful information that is related to the standard test properties such as tensile property, fracture toughness and ductile-brittle transition temperature. Some early work focused on obtaining empirical correlations between the transition temperature from the SP test and the fracture appearance transition temperature (FATT) and ductile-brittle transition temperature (DBTT) from the Charpy test [4-9]. More recently, Foulds et al. proposed an analytical method for directly measuring fracture toughness from the small punch test by calculating local strain energy in the SP specimen [10]. But these empirical and analytical approaches for predicting the standard properties have been mostly made for the unirradiated materials while the application for the irradiated materials to predict TTS by irradiation is limited.

In the present work, a small punch (SP) test was performed to evaluate the ductile-brittle transition temperature shift by neutron irradiation in reactor pressure vessel (RPV) forging steels. SP transition temperature ( $T_{SP}$ ) determined from SP energy transition curves were compared to the standard transition temperature from the Charpy test and the Master Curve fracture toughness test in accordance with the ASTM standard E1921.

### MATERIALS & TEST PROCEDURES

The materials selected for this study were four SA508 Cl.3 steels with different manufacturing processes, one SA508 Cl.2 steel and its weld using the Linde 80 flux, and SA533B Cl.1 steel (JRQ). Small punch samples were taken from the 1/4t thickness location of the vessels and machined to a 10x10x0.5mm dimension. Table 1 shows the standard fracture properties for the materials studied here. Irradiation was carried out in the research reactor at KAERI (HANARO) and NRI at 290°C to  $1.2\times$  and  $4.5\times 10^{19}$  n/cm<sup>2</sup> for the four SA508 Cl.3 steels.

Table. 1 The chemical compositions and the standard fracture properties for the RPV steels studied in the present work.

Types	ID	Chemistry (wt%)				USE (J)	T <sub>68J</sub> (°C)	T <sub>41J</sub> (°C)
		Ni	Cu	P	C			
SA508C1.3	KFY3	0.78	0.06	0.008	0.18	239	7.2	-3.3
	KFY4	0.78	0.06	0.007	0.19	281	-9.4	-21.5
	KFU4	0.86	0.03	0.006	0.18	282	-28.5	-24.6
	KFY5	0.92	0.03	0.007	0.21	267	-34.0	-44.7
	U4W	0.13	0.03	0.011	0.08	300	-9.4	-24.6
SA533B1	JRQ	0.84	0.14	0.017	0.18	207	-5.9	-19.6
SA508C1.2	K1	0.73	0.07	0.010	0.22	238	-28.1	-38.0
Linde 80 flux weld	K1W	0.61	0.23	0.012	0.10	91	28.9	-14.6

Fig. 1 shows the schematic illustration of the small punch test jig. Two thermocouples were inserted into the specimen holder and lower die to monitor specimen temperature and a LVDT gauge attached on the upper push rod measured punch displacement. The test was conducted at a constant crosshead speed of 0.1mm/sec in an environment chamber cooled with liquid nitrogen and the load-displacement curve was recorded for each sample. SP energy was obtained by calculating the area under the load-displacement curve to fracture. At each temperature in the transition range, 6 samples were tested and the mean value of the measured SP energies was determined by applying two-parameter Weibull statistics [8].

## RESULTS & DISCUSSIONS

### 1. Small Punch Test Results for the Unirradiated Materials

Fig. 2 shows the SP energy curves for the unirradiated materials. From the curves, the SP energy transition temperatures (T<sub>SP</sub>), which are defined by the midpoint between the upper and lower energy levels, are determined. Fig. 3 compares the T<sub>SP</sub> to the Charpy index temperatures (T<sub>41J</sub>, T<sub>68J</sub>). From the other reports, the empirical correlation between T<sub>SP</sub> and a ductile-brittle transition temperature (DBTT) from the Charpy test (T<sub>CVN</sub>) is generally given by

$$T_{CVN} (K) = \alpha T_{SP} (K),$$

where  $\alpha$  is a mechanical correlation factor. The different material and testing configuration yield different  $\alpha$  value. Unfortunately, the value has not been available for Mn-Mo-Ni low alloy steels used for nuclear RPV yet. The data in Fig. 3 yields  $\alpha=2.30\sim 2.75$  for T<sub>SP</sub>-T<sub>41J</sub> and  $\alpha=2.40\sim 2.92$  for T<sub>SP</sub>-T<sub>68J</sub>. Good correlations are found. But, the upper shelf

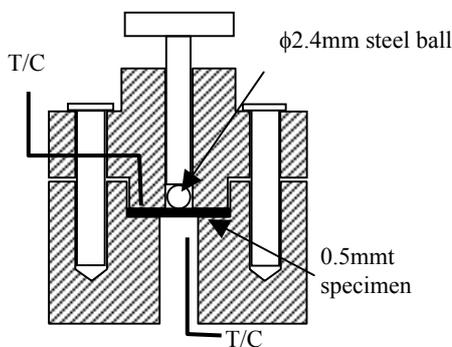


Fig. 1. The schematic illustration of the small punch test jig.

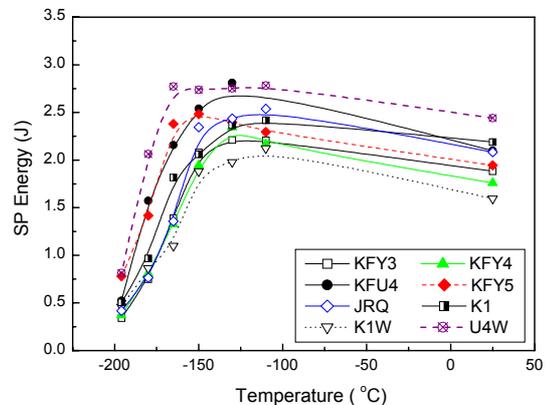


Fig. 2. Comparison of SP energy curves for the RPV steels.

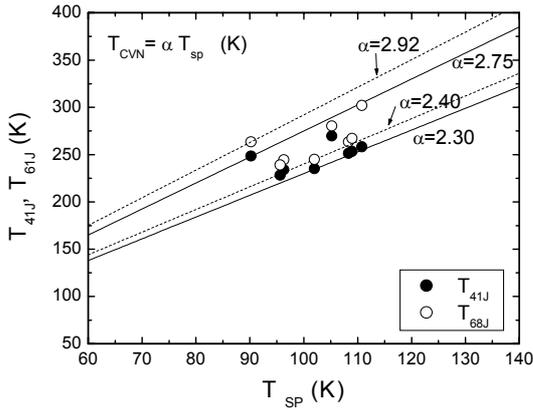


Fig. 3. Charpy index temperatures vs.  $T_{SP}$ .

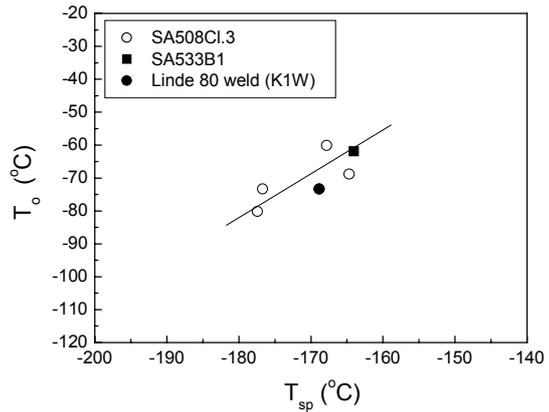


Fig. 4. Master curve transition temperature ( $T_0$ ) vs.  $T_{SP}$ .

energy levels in the SP test are not well matched with those from the Charpy test. Especially, the extremely low Charpy upper shelf energy level in Linde 80 weld does not correspond well to the SP test result.

Fig. 4 shows the correlation between  $T_{SP}$  from the SP test and  $T_0$  from the master curve fracture toughness test using PCVN specimen. One can see a meaningful linear correlation between them like a comparison to the Charpy data.

As is noted in the previous section, we tested 6 samples at each temperature in the transition region and determined the mean value at each temperature by using statistical analysis based on the Weibull distribution. In case of the irradiated sample testing, there exists limitation in the amount of testing material. Thus it is necessary to know the minimum number of testing samples at each temperature to give a statistically reliable result to the  $T_{SP}$  determination. For this, we took different sample sizes ( $n$ ) from the list arranged in the testing order, and calculated the mean value for each sample size and determined  $T_{SP}$ . Fig. 5 shows the change of the  $T_{SP}$  with different sample size. Above  $n=4$ ,  $T_{SP}$  is saturated to the nearly constant value. From this result, we determined 4 as a minimum sample number for yielding a reliable  $T_{SP}$  value.

## 2. Small Punch Test Results for the Irradiated Materials

Fig. 6 shows the typical changes of the load-displacement curves at  $-150$  and  $-180$  °C in the transition region after irradiation for KFY4 steel. As the fluence level increases, the load and displacement levels at the fracture become lower while the curve slope is slightly steeper. Fig. 7 shows the SP energy changes as a function of the testing temperature for the four materials before and after irradiation. One can find a greater shift in the curves with higher neutron fluence levels. The  $T_{SP}$  values for the irradiated materials are compared with the Charpy  $T_{41J}$  in Fig. 8 together with the results for the unirradiated materials. Unfortunately, the SP specimens at the same fluence level with the

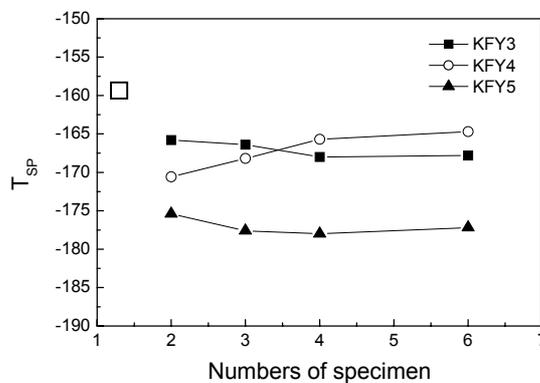


Fig. 5. The change of  $T_{SP}$  as a function of the specimen quantity.

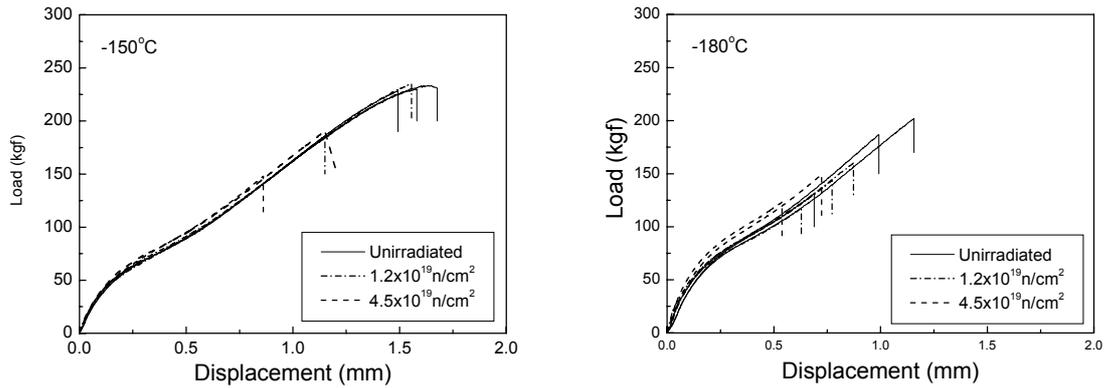


Fig. 6. SP load-displacement curves for KFY4 steels before and after irradiation.

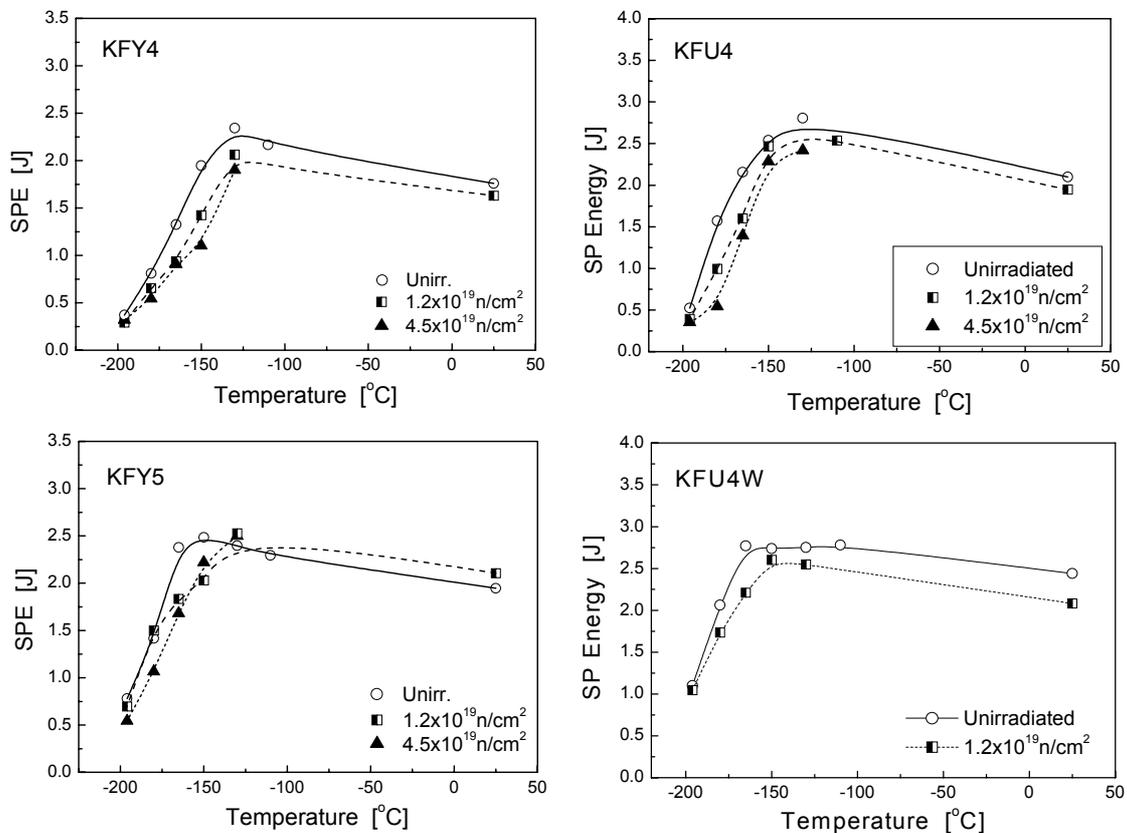


Fig. 7. The SP energy changes as a function of the testing temperature for the three materials before and after irradiation ( $E > 1\text{MeV}$ ): (a) KFY4, (b) KFU4, (c) KFY5, (d) KFU4W

Charpy specimens are not available for the materials except for KFY5 at this time. So we indicate those SP data as the ranges that are bounded by the  $T_{SP}$  values at the lower and higher fluence levels than that for the Charpy specimens. Though a direct comparison was not made, it provides a consistent linear correlation between  $T_{SP}$  and  $T_{41J}$ . The results for the irradiated materials are well within the  $\alpha$  range of the unirradiated materials. The total data in this study yields the following correlation:

$$T_{41J} \text{ (K)} = 2.43T_{SP} \text{ (K)}$$

Fig. 9 shows the  $T_{SP}$ - $T_0$  correlation including the results for the irradiated materials. It is still premature to make some conclusions from this small set of irradiation conditions. Nevertheless, they show a meaningful correlation

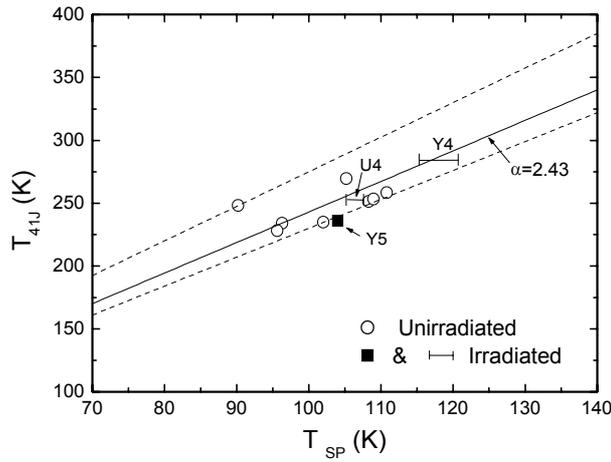


Fig. 8. The correlation between the  $T_{SP}$  and Charpy  $T_{41J}$  for the materials before and after irradiation.

between them for the irradiated materials. This correlation might come from the similarity in an underlying fracture mechanism though different testing methods. In brittle fracture, many researchers indicate the existence of the local cleavage fracture stress as a parameter governing fracture criterion [11-13]. So, it would be useful to compare the local fracture stresses of the two testing methods and further studies are under way.

## CONCLUSIONS

Small punch (SP) test technique was applied to evaluate the ductile-brittle transition temperature by neutron irradiation in RPV forging steels. SP transition temperatures ( $T_{SP}$ ), which is determined by the middle of the upper and lower SP energies, showed a good correlation with the Charpy index temperatures,  $T_{41J}$ . Including the data for the irradiated samples, the empirical correlation between them could be expressed as the following equation for RPV forging steels:

$$T_{41J} \text{ (K)} = 2.43T_{SP} \text{ (K)}$$

$T_{SP}$  also has a meaningful correlation with  $T_0$  values from the fracture toughness test in accordance with the ASTM standard E1921.

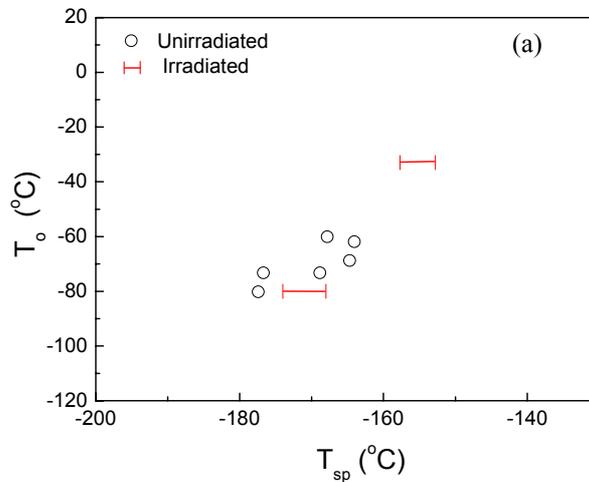


Fig. 9.  $T_{SP}$ - $T_0$  correlation

## ACKNOWLEDGEMENT

This work has been carried out as a part of the Materials Fracture Characterization and Toughness Improving Technology Development Project under the Nuclear R & D Program by MOST in Korea.

## REFERENCES

1. Wallin, K., Fracture of Engineering Materials & Structure, 1991, pp.83-88.
2. ASTM E 1921-97, "Standard Test Method for Determination of Reference Temperature,  $T_0$ , for Ferritic Steels in the Transition Range," ASTM, 1998.
3. ASME Code Case N-629, "Use of Fracture Toughness Data to Establish Reference Temperature for Pressure Retaining Materials for Section XI".
4. Bulloch, J.H., "Toughness losses in low alloy steels at high temperatures : an appraisal of certain factors concerning the small punch test", *Intl. J. Pressure Vessels and Piping*, Vol.75, 1998, pp.791-804
5. Baik, J.-M., Kameda, J. and Buck, O., "Small punch test of intergranular embrittlement of an alloy steel", *Scripta Metall.*, Vol.17, 1983, pp.1443-47.
6. McNaney, J., Lucas, G.E. and Odette, G.R., "Application of ball punch tests to evaluating fracture mode transition in ferritic steels", *J. Nucl. Mater.*, 179-181, 1991, pp. 429-433.
7. Misawa, T., Adachi, T., Saito, M. and Hamaguchi, Y., "Small punch tests for evaluating ductile-brittle transition behavior of irradiated ferritic steels", *J. Nucl. Mater.*, 150, 1987, pp. 194-202
8. Joo, Y.-H., Hashida, T. and Takahashi, H., "Determination of ductile-brittle transition temperature (DBTT) in dynamic small punch test", *J. Testing and Evaluation*, 20, 1992, pp. 6-14
9. Saucedo-Munoz, M.L., Matsushita, T., Hashida, T., Shoji, T. and Takahashi, H., "Development of a Multiple Linear Regression Model to Estimate the Ductile-Brittle Transition Temperature of Ferritic Low-Alloy Steels Based on the Relationship Between Small Punch and Charpy V-Notch Tests", *J. Testing and Evaluation*, 28, 2000, pp. 352-358.
10. Foulds, J.R., Woytowicz, J.R., Parnell, T.K. and Jewett, C.W., "Fracture toughness by small punch testing", *J. Testing and Evaluation*, 23, 1995, pp. 3-10.
11. Chen, J.H. and Wang, G.Z., "Study of mechanism of cleavage fracture at low temperature", *Metall. Trans.*, 23A, 1992, pp. 509-517.
12. Chen, J.H., Wang, G.Z., Zhu, L. and Gao, Y.Y., "Further study on the scattering of the local fracture stress and allied toughness value", *Metall. Trans.*, 22A, 1991, pp. 2287-2296.
13. Yang, W.J., Huh, M.Y., Roh, S.J., Lee, B.S., Oh, Y.J. and Hong, J.H., "Mechanical Behavior : The Effects of the Local Fracture Stress and Carbides on the Cleavage Fracture Characteristics of Mn-Mo-Ni Low Alloy Steels in the Transition Region", *J. Kor. Inst. Met. & Mater.*, 38, 2000, pp. 675-680.