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EVALUATION OF TOUGHNESS DEGRADATION BY SMALL PUNCH (SP) TESTS FOR NEUTRON IRRADIATED STRUCTURAL STEELS

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Abstract: The small punch (SP) test as one of the useful small specimen testing technique (SSTT) has been developed to evaluate the fracture toughness, ductile-brittle transition temperature (DBTT) and tensile properties for neutron irradiated structural materials. The SP tests using the miniaturized specimens of ϕ3 mm TEM disk and 10 mm^2 coupon were performed for six kinds of ferritic steels of F-82, F-82H, HT-9, JFMS, $2^1/_4$-1Mo and SQV2A. It was shown that the temperature dependence of SP fracture energies with scatter in miniaturized testing can give reliable information on the DBTT by use of the statistical analysis based on the Weibull distribution. A good correlation between the DBTT of the SP tests and that of the standard CVN test has been obtained for the various nuclear ferritic steels. The SP test was performed for cryogenic austenitic steels as a way of evaluating elastic-plastic fracture toughness, J_{IC}, on the basis of a universal empirical relationship between J_{IC} and SP equivalent fracture strain, $\bar{\varepsilon}_{qf}$. The SP testing using the neutron irradiated specimens of $2^1/_4$Cr-1Mo, F-82, F-82H and HT-9 steels was successfully applied and presented the neutron radiation induced changes on the DBTT, fracture toughness and tensile properties.

Keywords: small specimen testing technique, small punch (SP) test, DBTT, FATT, statistical analysis, Weibull distribution, fracture toughness, low activation ferritic steel, neutron irradiation, SCC

1. INTRODUCTION

Small specimen testing technique (SSTT) is absolutely necessary in fusion reactor material development, since facilities for irradiation tests usually have very limited space available for irradiation. Although several kinds of methods using miniaturized specimens have been proposed, certain kinds of mechanical properties, such as fracture toughness and ductile-to-brittle transition behavior in ferritic structural steels, are very difficult to measure with small and/or limited number of the neutron irradiated specimens. A small punch (SP) test system using miniaturized specimens has been well developed on the basis of the results of cooperative research by the Muroran Institute of Technology [1~4], JAERI [4~6], Tohoku University [6,7,8] and Iowa State University [9,10]. A recommended practice [4] for the SP test has been drafted in the JAERI-M 88-172 report [4]. As shown in Fig.1, an SP test system using miniaturized specimens has been well established to evaluate the ductile-

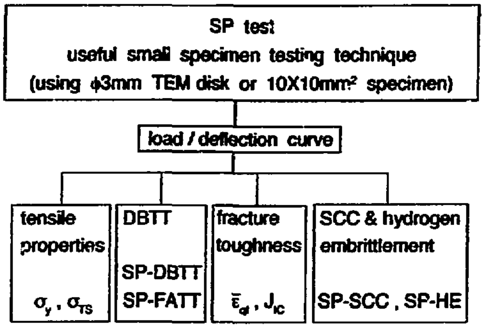

Fig.1 A progress of evaluation system based on small punch(SP) tests for material degradation in neutron irradiated structural alloys in nuclear technology.

brittle transition temperature (DBTT) for irradiated ferritic steels [1,3,5,6,9,10], the fracture toughness for austenitic [2] and ferritic [5~8,14,15] steels and advanced ceramics [15], the tensile properties [5,14,15], the susceptibility of stress corrosion cracking (SCC) and hydrogen embrittlement in candidate structural steels [11,12] and intermetallic compound [13].

In this paper, recent developments in SP testing have been reviewed, with the focus on the authors' work between MIT and JAERI.

2. EXPERIMENTAL

The materials used for the SP tests of DBTT evaluation were six kinds of normalized and tempered ferritic steels, i.e. two low activation ferritic steels of 8Cr-2W-V (F-82) and 8Cr-2W-V-Ta (F-82H), two conventional steels of 12Cr-1Mo-W-V (HT-9) and 9Cr-2Mo-1Ni-V-Nb (JFMS), two low alloy steels of $2^1/_4$-1Mo and long-term aged nuclear pressure vessel material SQV2A (SA533B cl.2). Chemical compositions and heat treatment conditions are summarized in Table 1. Fracture toughness and equivalent fracture strain by the SP tests were evaluated for neutron-irradiated ferritic structural steels of $2^1/_4$-1Mo, F-82 and F-82H, and four kinds of austenitic structural steels in nuclear fusion apparatus at cryogenic temperatures. Chemical compositions and mechanical properties of the austenitic steels are given in the previous paper [2]. Two kinds of specimens for the SP tests were fabricated, 10x10 mm^2 coupons, 0.5 [2,13,16] or 0.25 mm [1,5,11,12] thick and 3 mm in diameter TEM disks with 0.25 mm thickness [1,3,6]. Some of these specimens were cut from the standard Charpy V-notched (CVN) specimens which had already been tested. The SP tests were performed over a range of test temperatures from liquid nitrogen to 473 K [1,3,5,6,13,16] and at 4.2 K [2]. The SP fracture energy necessary for macroscopic cracking was taken as the area under the load versus deflection curve up to the sudden drop of load. The equivalent fracture strain, $\bar{\varepsilon}_{qf}$, for evaluation of elastic-plastic fracture toughness, J_{Ic}, was obtained [2,5,6]. The apparatus and SP testing were described previously [1~4]. Neutron irradiation was performed in Japan Research Reactor-2 (JRR-2) to a neutron fluence of 2 x 10^{23} n/m^2 (E>1 MeV) at 573 K for $2^1/_4$-1Mo steel [5] and a neutron fluence of 1.2 x 10^{24} n/m^2 at 600 K for F-82, F-82H and HT-9 steels [6], respectively. The SP tests on irradiated specimens were conducted in the Tokai Hot Laboratory using an Instron-1361 creep-fatigue testing machine in the temperature range from 113 K to 300 K. A hardened steel ball of 2.4 mm diameter was used for punching coupon type specimens, and a 1 mm diameter ball for 3 mm diameter disk type specimens [4]. All SP tests for the irradiated specimens were performed at a crosshead speed of 8.3 x 10^{-3} mm/s.

Table 1 Chemical compositions (mass%) and heat treatments of the materials used

Alloy	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	W	V	Ta	Nb	N
F-82	0.100	0.17	0.49	0.003	0.002	0.05	7.52	tr.	-	2.19	0.19	-	-	0.0018
F-82H	0.093	0.09	0.49	0.005	0.001	0.01	7.65	tr.	-	1.98	0.18	0.038	-	0.0019
HT-9	0.20	0.21	0.52	0.008	0.010	0.52	12.24	1.03	-	0.48	0.29	-	-	0.0063
JFMS	0.05	0.67	0.58	0.009	0.006	0.94	9.85	2.31	-	-	0.12	-	0.06	0.0056
$2^1/_4$Cr-1Mo	0.14	0.08	0.54	0.09	0.09	0.12	2.36	1.04	0.09	-	-	-	-	-
SQV2A	0.15	0.25	1.32	0.008	0.002	0.65	0.12	0.49	0.007	-	-	-	-	-

Alloy	Normalizing	Tempering	Microstructure
F-82	1183 K 0.5h	983 K 2h	Tempered martensite
F-82H	1313 K 0.5h	1013 K 2h	Tempered martensite
HT-9	1273 K 2h	(1043,973,873,773)K 2h	Tempered martensite plus δ-ferrite
JFMS	1323 K 0.5h	1048 K 1h, 773 K 1000h	Ferrite/tempered martensite
$2^1/_4$Cr-1Mo	1183 K 5h	923 K 6h + 958 K 22h(post weld H.T)	Bainite with a small amount of pro-eutectoid ferrite
SQV2A	As-received	888 K 45h (SR) SR + 723 K 3000h	

3. RESULTS AND DISCUSSION

3.1 Load-deflection Curve

3.1.1 Tensile Properties

Typical load-deflection curves for $2^1/_4$-1Mo steel before and after irradiation are shown in Fig.2 [5]. The load-deflection curves shifted upwards after irradiation, indicating hardening caused by irradiation. Changes in tensile properties and SP-related values caused by irradiation are compared to show the correlation among these values. Figures 3(a) and (b) [5] show the strength parameter correlation in terms of yield (0.2% off-set) and ultimate tensile stress, P_y/t_0^2 and P_{max}/t_0^2 were selected as the SP-related values corresponding to yield and ultimate tensile stress, where t_0 is the initial thickness of the SP specimen and P_y and P_{max} are defined as shown in Fig.2. There is a clear correlation among these strength parameters, indicating that irradiation caused shifts along this band.

By contrast, ductility parameters in terms of elongation from tensile tests did not have a good correlation with any of the SP-related values even though some corrections were attempted from the point of compliance of the testing apparatus. Values of the ratio δ_u/t_0 or δ^*/t_0 (see definition in Fig.2) as a function of corresponding tensile elongation were apparently not correlated with each other [5]. Nevertheless, it is to be noted that the changes in the ratio δ^*/t_0 and elongation caused by irradiation coincide well with each other. That is, slopes of the lines connecting the data points on the same material before and after irradiation were all nearly equal.

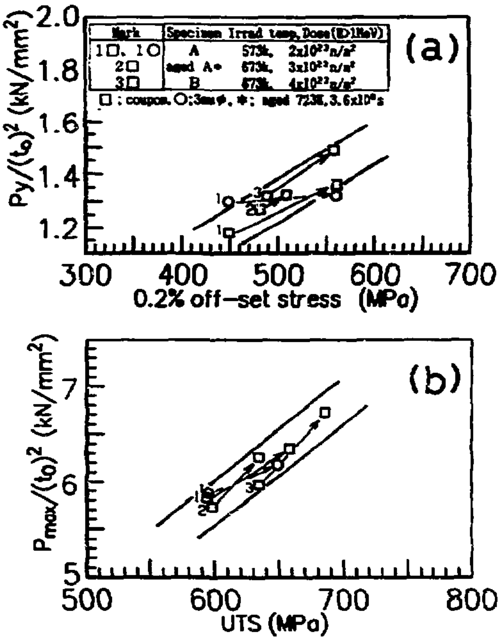

Fig.3 Strength parameter correlation for the yield 0.2% off-set (a) and the ultimate tensile strength (b). The definitions for the SP test-related parameters are shown in Fig.2.

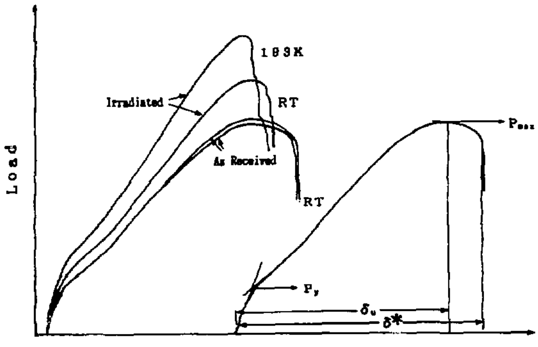

Fig.2 Schematic illustrations of load-deflection curves for $2^1/_4$-1Mo steel before and after irradiation (573 K, 2×10^{23} n/m^2, E > 1 MeV), and designation of the SP-related mechanical property parameter.

3.1.2 Fracture Toughness

Basic idea to expect the value of elastic-plastic fracture toughness, J_{IC}, from a specimen as small as ϕ3 mm TEM disk or 10x10 mm^2 size lies in the fact the J_{IC} should have a clear relationship with biaxial equivalent fracture strain, $\bar{\varepsilon}_{qf}$. Mao et al. [7,8] tested for ferritic steels at room temperature and authors [2] tested for austenitic steels at 4.2, 77 and 293 K assumed the following equations to calculate $\bar{\varepsilon}_{qf}$.

$$\bar{\varepsilon}_{qf} = \ln\,(t_0\,/\,t^*) \qquad (1)$$
$$= \beta\ (\delta^*/\,t_0)^n \qquad (2)$$

where t_0 is the initial thickness, t^* is the minimum thickness at the fracture portion, β is ~ 0.09 [2,7] and n ~ 2 [2,7] for both ferritic [7] and austenitic [2] steels, and δ^* is a deflection at which a sudden load drop occurs, as shown in Fig.4 [2]. Figure 5 [2] shows a regression line for ln ln(t_0/t) versus ln(δ/t_0) using the data for fractured *and unbroken specimens for four kinds of* stable austenitic steels at different temperatures. Parameters β and n can be obtained from the this linear regression. The calculation of $\tilde{\varepsilon}_{qf}$ using the equation (1) needs measurement of the specimen thickness after fracture. The simplified determination can be made from the load-deflection curve with the equation (2).

Correlation between the J_{IC} including *both valid and invalid data and the practical* equivalent strain $\tilde{\varepsilon}_{qf}$ estimated from the δ^* using the above parameters in stable austenitic steels is shown in Fig.6 [2]. Since the J_{IC} of ductile austenitic steels obtained at room temperature is invalid, linear correlation is rather poor. However, the regression coefficient of 686 kJ/m^2 has substantial meaning for a qualitative estimation of the relative change in fracture *toughness due to neutron irradiation from* the SP test. Figure 7 [7,8] shows a linear dependence of J_{IC} on fracture strain $\tilde{\varepsilon}_{qf}$ for various ferritic steels. For practical application of the SP test to the estimation of J_{IC} above room temperature, an extended empirical relation among δ^*, $\tilde{\varepsilon}_{qf}$ and J_{IC} is proposed [2]. From a practical point of view, a way of the J_{IC} value evaluation by SP test using ϕ3 mm TEM disk is very useful for the semiquantitative estimation of the relative change in fracture toughness due to heavy neutron irradiation [5,6].

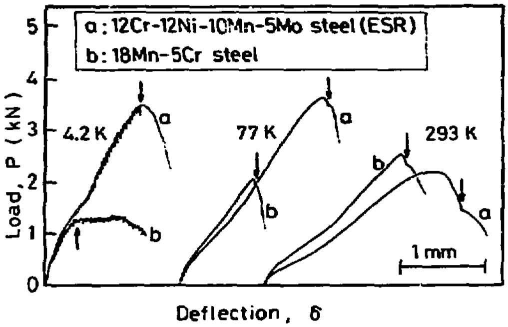

Fig.4 Load versus deflection curves obtained from the SP tests for two austenitic steels, including 12Cr-12Ni-10Mn-5Mo-N alloy of manganese- and nitrogen-strengthed stable austenitic stainless steel which satisfied the the requirements of the JAERI box for the Fusion Experimental Reactor (FER) superconducting magnet structural steels at liquid helium (4.2 K). Arrows indicate the maximum deflection at fracture, δ^*.

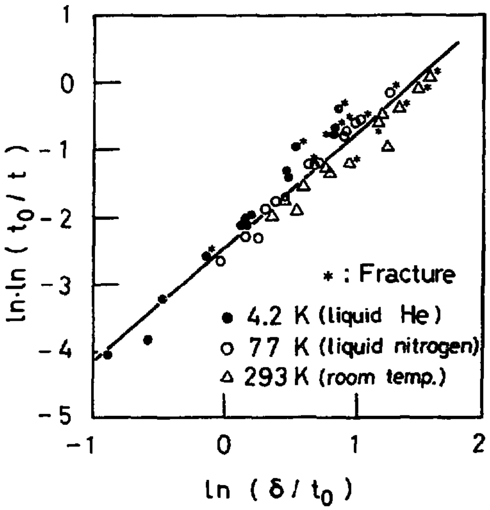

Fig.5 Relationship between ln ln(t_0/t) and ln(δ/t_0) for various austenitic steels, after the SP tests.

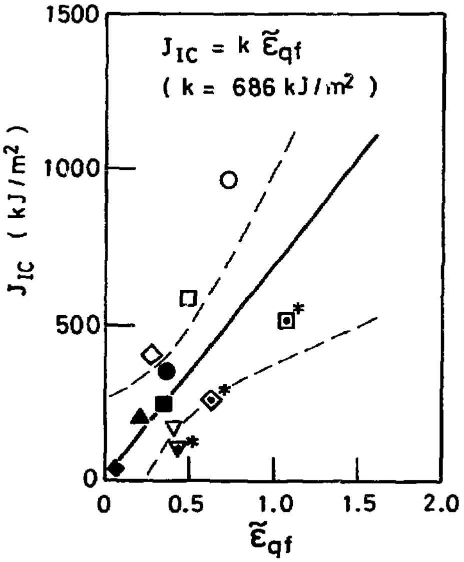

Fig.6 An extended plot of the J_{IC} values including invalid J_{IC} versus the $\tilde{\varepsilon}_{qf}$ for various austenitic steels at test temperatures of 4.2, 77 and 293 K. The short-dashed lines indicate the 95% confidence limit, while the asterisks identify the invalid J_{IC} values at 293 K.

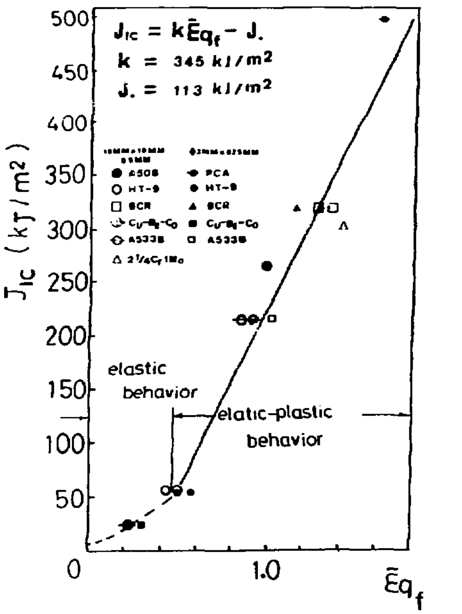

Fig.7 A linear dependence of measured J_{Ic} on $\bar{E}_{qf}$ in elastic-plastic behavior for various ferritic steels at room temperature.

3.2 Temperature Dependence of SP Energy and DBTT Evaluation

3.2.1 Ductile-Brittle Transition and DBTT

SP energy was defined as the energy consumed up to the onset of macrocracking, which correspond to the area under the load versus deflection curve up to a deflection δ^*. With decreasing test temperature, the maximum load gradually increases until a sudden decrease is observed to accompany a sudden decrease of deflection, as shown in Fig.8 [1]. Hence, as shown in Fig.9 [1], SP energy falls drastically at a certain temperature as the temperature decreases. This transition temperature (SP-DBTT) has approximately linear relation with the DBTT measured by the standard Charpy V-notched test (CVN-DBTT), that is,

$$\text{SP-DBTT} = \alpha \times \text{CVN-DBTT} \qquad (3)$$

where α is the correlation coefficient. Figure 10 shows an empirical correlation between the DBTT of the CVN test and that of the SP test for the miniaturized specimens of 10 mm^2 coupon and ϕ3 mm TEM disk in various kinds of ferritic steels by authors [1,3,5,16], including hot specimens of 2$^1/_4$-1Mo steel [5].

Recently, we have observed a temperature dependence of the cleavage brittle fracture fraction on the fracture surface of the SP specimens of SQV2A steel. Figure 11 [16] shows a clear ductile to brittle transition in fracture surfaces. The fracture appearance transition temperature by the SP test (SP-FATT) can be determined as the temperature corresponding to the 50 percent point of brittle fracture in fracture surface, observed throughout the whole thickness at the position of fracture initiation. A correlation between the SP-FATT and the FATT obtained from the CVN impact test (CVN-FATT) has been confirmed in our laboratory [16].

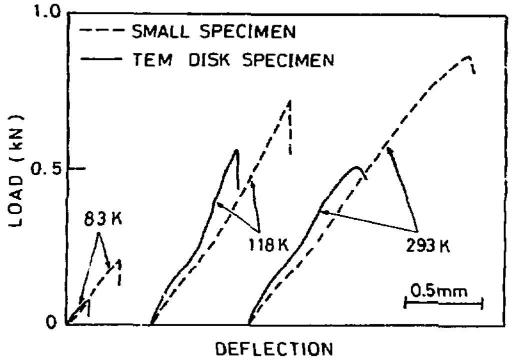

Fig.8 Examples of load versus deflection curves for SP tests of small (10x10x0.25 mm^3) and TEM disk (3 mm in dia. and 0.25 mm thick) specimens on HT-9 steel tempered for 2 h at 973 K, at various temperatures.

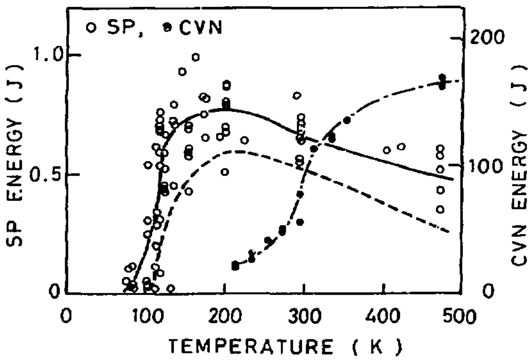

Fig.9 Temperature dependence of SP energies and mean value line in a Weibull distribution versus test temperature for HT-9 steel, tempered for 2 h at 973 K. Charpy V-notch (CVN) energy measurement curve is also shown.

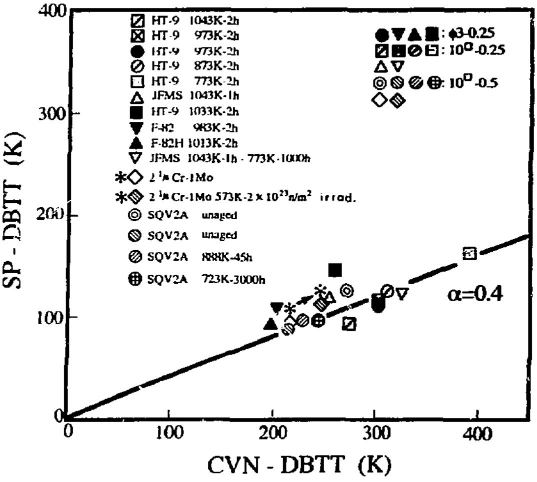

Fig.10 Correlation of the DBTT between standard CVN and SP tests for various ferritc steels.

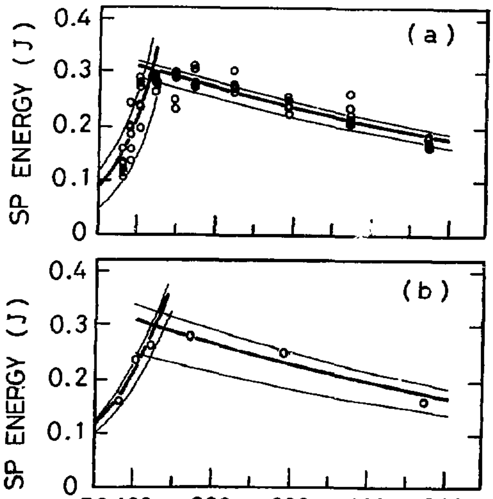

Fig.12 Examples of the temperature dependence of SP fracture energy estimates based on the data partitioning method for TEM disk specimens of F-82 steel: (a) all the measured every six data at ten test temperatures, (b) a single datum at six test temperatures. The middle solid lines show the estimated E_0 in two separated regions. A set of parallel thin lines indicates the lower- (5% failure probability) and upper- (95% failure probability) bound fracture energies.

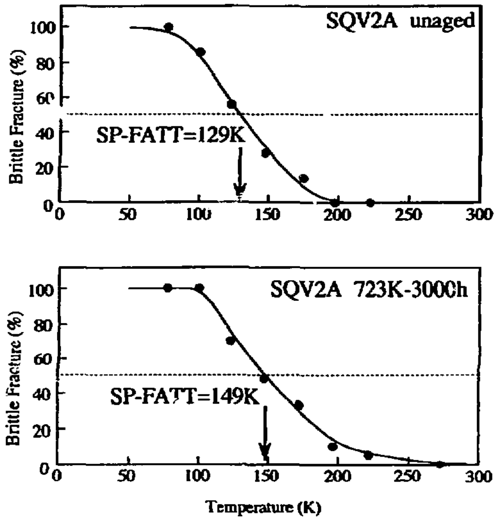

Fig.11 Temperature dependence of brittle fracture surface and shift of FATT for SQV2A steel with aging at 723 K for 3000 h, in SP test.

3.2.2 Reliable DBTT Determination Based on the Statistical Analysis

There is a SP-energy scatter caused by unavoidable microstructural heterogeneity to cracking in a miniature specimen of engineering alloys, as shown in Fig.9. The authors [1,3] have succeeded in the temperature dependence of SP fracture energies with scatter in miniaturized testing could give reliable information on the DBTT by use of the statistical analysis based on the Weibull distribution. We adopted the Weibull distribution of the following equation to express the probability distribution of SP energy,

$$F(x) = 1 - \exp[-(x/E_0)^m] \qquad (4)$$

where m (shape parameter) and E_0 are distribution parameters. The detailed procedure is described in the previous papers [1,3]. Figure 12 [3] shows the examples of the temperaure dependence of SP energy estimates based on the data partitioning method for TEM disk specimens of F-82 steel. Figure 12(b) shows an estimation required from a few data on the background of smaller irradiation volumes. The practical determining procedure of the minimum quantity of irradiated specimens for the reliable DBTT evaluation prior to irradiated material SP testing in the hot cell, with the aid of statistical analysis has been summarized in a flow diagram of Fig.13 [3].

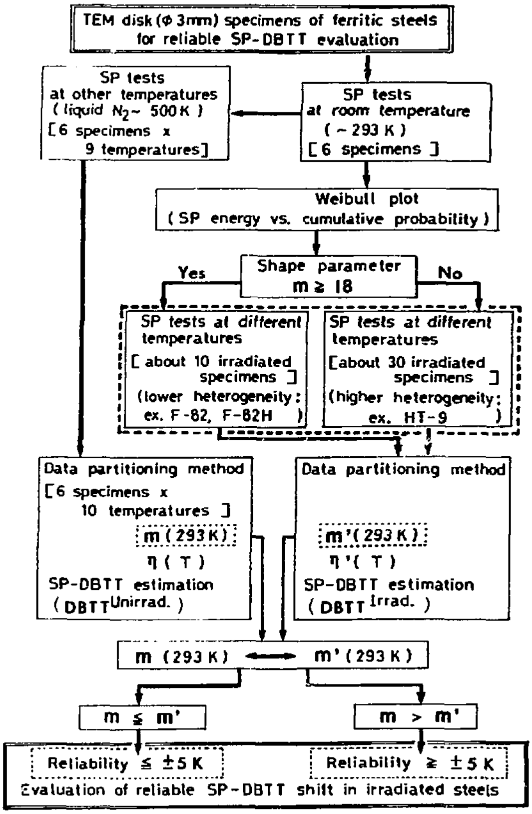

Fig.13 Flow diagram for the determination of the minimum quantity of irradiated specimens prior to irradiated material SP-DBTT testing. The experiment concerning the irradiated materials is surrounded by a thick broken line.

3.2.3 *Small Punch DBTT* Testing *Using Neutron-Irradiated Specimens*

Figure 14 shows the SP energy transition curves for neutron-irradiated coupon specimens of normalized and tempered $2^1/_4$-1Mo ferritic steel [5]. The DBTT shift was about 17 K before and after irradiation. By contrast,

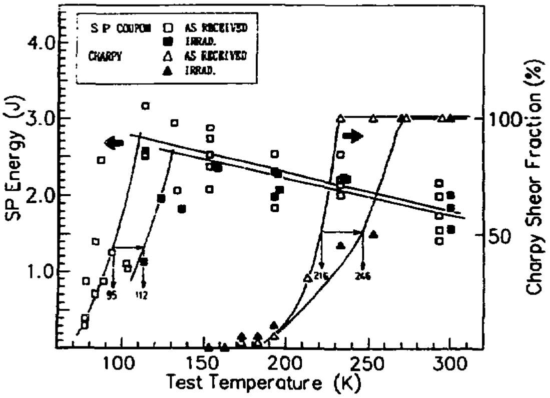

Fig.14 SP energy and Charpy transition curves before and after irradiation at 573 K to 2 x 10^{23} n/m^2 (E > 1 MeV).

the DBTT shift determined from the ductile-brittle transition curve in Charpy tests was 30 K. This DBTT shift before and after neutron irradiation is plotted in Fig. 10, and lies in the SP-DBTT/CVN-DBTT correlation obtained from the various kinds of cold specimens. All of these seem to show reasonable agreement. Besides the DBTT correlation, an important fact is that the fracture appearance changed clearly after the irradiation, and the load/deflection curve changed accordingly. That is, for the radiation-embrittled specimens, the load fails in a stepwise manner after the maximum load as shown in Fig.2, possibly due to crack branching. This indicates the possibility of evaluation of radiation embrittlement based on fractography-oriented analysis, such as Fig.11, together with the analysis of load/deflection curve, DBTT, other mechanical properties and microstructures.

A certain number of SP test on neutron-irradiated specimens using the 3 mm diameter TEM disks has been successfully done for the determination of the reliable DBTT [6]. Only 5 to 6 TEM disk specimens were tested at temperatures from 113 K to 300 K for low activation ferritic steels of F-82 and F-82H, according to the test procedure to determine the DBTT with the aid of statistical analysis by authors [3]. The result is encouraging the idea that the determination of DBTT would possible with limited number of irradiated specimens.

Incorporating a property-related correlation with SP testing will be a future subject when we will deal with material subjected to a higher dose.

3.3 SCC and Hydrogen Embrittlement Studies

A newly developed SP test technique combined with electrochemical measurements using miniaturized specimens in a high temperature and high pressure aqueous solution was successfully demonstrated to be useful for the evaluation of the stress corrosion cracking (SCC) susceptibility of Type 304 austenitic and HT-9 ferritic stainless steels [11,12]. The SP test was applicable for the evaluation of hydrogen embrittlement susceptibility of HT-9 ferritic steel [12] and Co_3Ti intermetallic compound [13] as a function of crosshead speed. The SP-SCC testing method is suitable for the rapid screening of irradiated miniaturized specimens for the evaluation of irradiation-assisted SCC (IA-SCC) susceptibility and corrosion resistance of fusion and fission reactor structural alloys in a water-cooled environment with irradiation.

4. CONCLUSIONS

The state of the present art for extracting toughness degradation related mechanical properties from the miniaturized specimens of ϕ 3 mm TEM disk and 10 mm^2 coupon by the SP test and advanced results on neutron-irradiated specimens were reviewed and discussed, on the basis of the authors' recent development by cooperative research between MIT and JAERI. It was concluded that the SP test is a very useful way of small specimen testing for the evaluation of the toughness changes, such as fracture toughness J_{IC}, DBTT, FATT, 0.2% off-set stress, ultimate tensile strength, susceptibility of SCC and hydrogen embrittlement, due to neutron irradiation and/or long-term aging in nuclear energy and fusion structural material program.

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