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Fusion Engineering and Design 81 (2006) 1077–1084
Fusion Engineering and Design
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Mechanical properties of small size specimens of F82H steel

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Received 1 February 2005; received in revised form 12 August 2005; accepted 12 August 2005
Available online 24 January 2006

Abstract

Small specimen test technology (SSTT) has been developed to investigate mechanical properties of nuclear materials. SSTT has been driven by limited availability of effective irradiation volumes in test reactors and accelerator-based neutron and charged particle sources. In this study, new bend test machines have been developed to obtain fracture behaviors of F82H steel for very small bend specimens of pre-cracked t/2-1/3CVN (Charpy V-notch) with 20 mm length and deformation and fracture mini bend specimen (DFMB) with 9 mm length and disk compact tension of 0.18DCT (disk compact tension) type, and fracture behaviors were examined at 20 °C. The effect of specimen size on ductile–brittle transition temperature (DBTT) of F82H steel was examined by using 1/2t-CVN, 1/3CVN and t/2-1/3CVN, and it was revealed that DBTT of t/2-1/3CVN and 1/3CVN was lower than that of t/2-CVN. DBTT behaviors due to helium and displacement damage in F82H-std irradiated at about 120 °C by 50 or 100 MeV He ions to 0.03 dpa were also measured by small punch tests.

Keywords: Fracture toughness; Radiation effects; Low activation materials; Ductile–brittle transition; Experimental techniques

1. Introduction

Small specimen test technology (SSTT) has been developed to investigate mechanical properties of nuclear materials. SSTT was driven by limited availability of effective irradiation volumes in test reactors and accelerator-based neutron and charged particle sources [1–5]. The most recent efforts in fracture SSTT have been pursued within the framework of the master curves-shifts method proposed by Odette et al. for bcc alloys such as ferritic/martensitic steels currently being considered as the first candidate for fusion reactor structures [6,7]. The master curves-shifts represent a significant extension and modification of recent developments in measuring cleavage initiation toughness for heavy section component integrity assessments. The

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0920-3796/$ – see front matter
doi:10.1016/j.fusengdes.2005.08.072

success in utilizing small specimens to obtain meaningful and useful fracture toughness information is leading to an even more aggressive approach to reducing fracture specimen sizes. A variety of tests have been devised to extract mechanical property data from existing small volume specimens, such as tensile, low and high cycle fatigue, fracture toughness, fatigue crack growth, pressurized tubes, notched and pre-cracked impact specimens and 3 mm diameter disks. A subset of these specimens and techniques has been tentatively selected as candidate for materials response verification in the International Fusion Materials Irradiation Facility (IFMIF), which is a D-Li-based high-energy neutron source currently undergoing conceptual design [8].

In this study, we have examined the fracture toughness of F82H steel using three type specimens of 0.18DCT (disk compact tension), pre-cracked t/2-1/3CVN (Charpy V-notch), which is denoted as t/2-1/3PCCVN, with 20 mm length and deformation and fracture mini bend specimen (DFMB) with 9 mm length. The 0.18DCT and 1/3CVN specimen is recently used for a standard fracture specimen of the neutron irradiation study in reduced-activation ferritic (RAF) steels. But the volumes of 0.18DCT and 1/3CVN specimens are not so small in the space of irradiation facility such as IFMIF. It is very important to reduce the specimen volume for the performance of irradiation dose. The blanket structure of fusion reactors will be composed of RAF steel plates and pipes with different thickness, and we should check the dependence of specimen size on the properties of fracture toughness. The t/2-CVN specimen reduced the width of 1/3CVN specimen by half, and the DFMB specimen reduced the width and length of 1/3CVN by about half.

A new bend test machine has manufactured to obtain fracture behavior for very small bend specimens of t/2-1/3PCCVN and DFMB. The fracture behaviors of F82H steel with different type of shapes have been examined. The types of specimens used in this study are 0.18DCT, 1/3CVN, t/2-1/3CVN, t/2-1/3PCCVN, DFMB and small punch (SP) specimens. The purpose of this study is: (1) to manufacture fracture toughness testing machines for the small specimens of DFMB and t/2-1/3PCCVN and to check the fracture behaviors of F82H-std steel at RT and (2) to examine the effect of specimen size on ductile–brittle transition temperature (DBTT) of F82H-std steel.

2. Fabrication of DFMB, t/2-1/3PCCVN and 0.18DCT specimens

The fatigue pre-cracks of 0.18DCT, t/2-1/3PCCVN and DFMB specimens were induced by using a fatigue testing machine of Shimazu Lab-5u. The 0.18DCT specimens (12.5 mm diameter and 4.63 mm thickness) were machined in the T–L orientation so that crack propagation occurred parallel to the rolling direction. Fatigue pre-cracking was performed at room temperature in a condition of crack length to specimen width ratio (a/W) of approximately 0.46. This was followed by side-grooving on each side to the depth of ~10% of specimen thickness. The length of fatigue pre-crack extension of 0.18DCT specimen was about 1.3–1.4 mm. The applied load at the first step was changed between 108 and 1079 N at 40 Hz and it was changed between 88 and 883 N at the next step. In the t/2-1/3PCCVN and DFMB specimens, the pre-crack was induced in the plate shape with V-notch and U-notch, respectively, and the size of the plates was 20 mm × 20 mm square and 3.3 mm in thickness. The depth and angle of V-notch in the t/2-1/3PCCVN were 0.51 mm and 30°, respectively. In the DFMB specimen, U-notch was adopted to reduce the notch region by using a 0.15 mm wire cutter, and the depth and width of U-notch was about 0.5 mm and about 0.2 mm, respectively. The load of the plate for the preparation of t/2-1/3PCCVN and DFMB specimens was changed from 294 to 2942 N at 40 Hz. After the pre-crack procedure, the plate was sliced to about 1.7 mm in thickness by wire cutting. The lengths of the pre-crack of t/2-1/3PCCVN and DFMB specimens were about 0.9 mm and about 0.3 mm, respectively. The ratio of crack length to specimen thickness, a/W, for the DFMB and t/2-1/3PCCVN specimens was controlled to the values from 0.40 to 0.45. The chemical compositions of the specimens in this study are given in Table 1.

3. Fracture toughness testing

3.1. Fracture toughness testing of DFMB and t/2-1/3PCCVN

Fig. 1 shows a new bend test machine, which is manufactured to obtain fracture behavior for very small bend specimens of t/2-1/3PCCVN with 20 mm length

Table 1
Chemical compositions of the specimens used in this study (wt%)

Materials	N	C	Si	Mn	P	S	Cr	W	V	Ta
F82H-std (IEA)	0.007	0.09	0.07	0.10	0.003	0.001	7.82	1.98	0.19	0.04
F82H-std (low N)	0.0023	0.099	0.11	0.10	0.007	0.001	7.92	1.97	0.18	0.05

(W: 3.3 mm, H: 1.65 mm) and DFMB with 9 mm length (W: 1.65 mm, H: 1.65 mm). The displacement rates of cross head in this machine can be changed from 0.01 to 100 mm/min. The temperature can be controlled by the amounts of vapor of liquid nitrogen with high pressure and electric heater, and it can be changed from −196 to 300 °C. The deviation of temperature is within about 0.5 °C. Fig. 2(a and b) show DFMB and t/2-1/3PCCVN specimens set in the stage of the bend test machine, respectively. The scale of 0.5 mm distance was set in the back of specimen setting position. The adjustment of specimen position in the specimen stage is controlled by a small micrometer, which is set in the machine.

The position of the load cell can be measured by using a linear gauge (Mitutoyo, LGF sereies) and it is also controlled by a feedback control system for the position. The accuracy of the position for linear gauge is ±0.5 μm. The position of the specimen on the specimen stage equipped with a scale can be adjusted by using a small μ-meter instrument. The cross head is dropped gradually to the center of specimen with V-

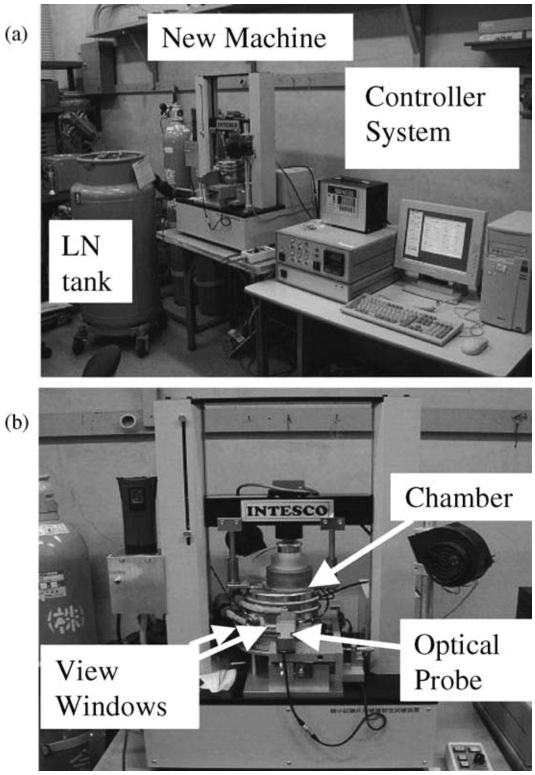

Fig. 1. A new bend test machine, which is manufactured to obtain fracture behavior for very small bend specimens of pre-cracked 1/3CVN with 20 mm length and DFMB with 9 mm length: (a) configuration of the machine and (b) chamber and the optical probe.

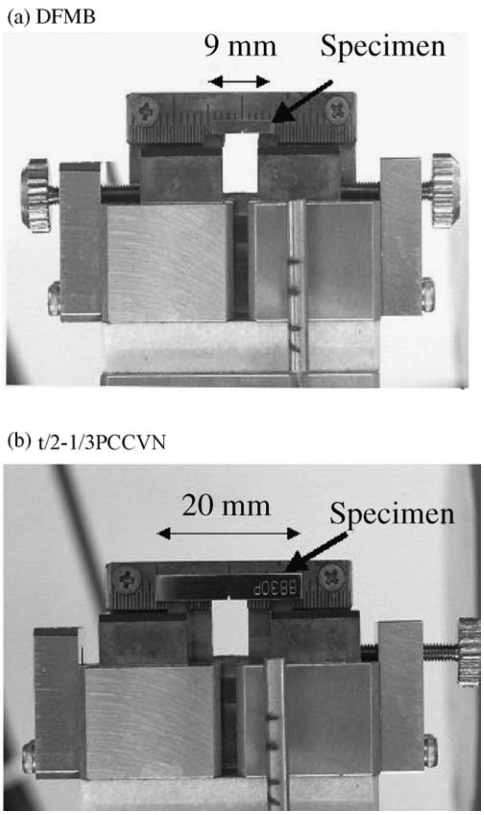

Fig. 2. (a) DFMB specimen and (b) t/2-1/3PCCVN specimen of F82H-std (low N) set in the stage.

notch. The displacement of the specimen is measured exactly by an optical probe (Keyence, LS-7030T) from the view port set in the chamber as shown in Fig. 1(b), and the accuracy for the displacement of the specimen is within ±0.15 μm.

Fig. 3(a and b) is load–displacement curves tested at 20 °C in the DFMB and t/2-1/3PCCVN F82H-std specimens, respectively. The cross head of 0.1 mm/min was selected under the unloading compliance method. The fracture toughness tests for compact and three-point bend specimens was performed under the guidelines of the ASTM E 813–89 and E 1820–99a. Fracture toughness of DFMB and t/2PCCVN at RT was about 170 and 230 MPa m$^{1/2}$, respectively. These values were smaller than the value of 0.18DCT specimen as described in Section 3.2.

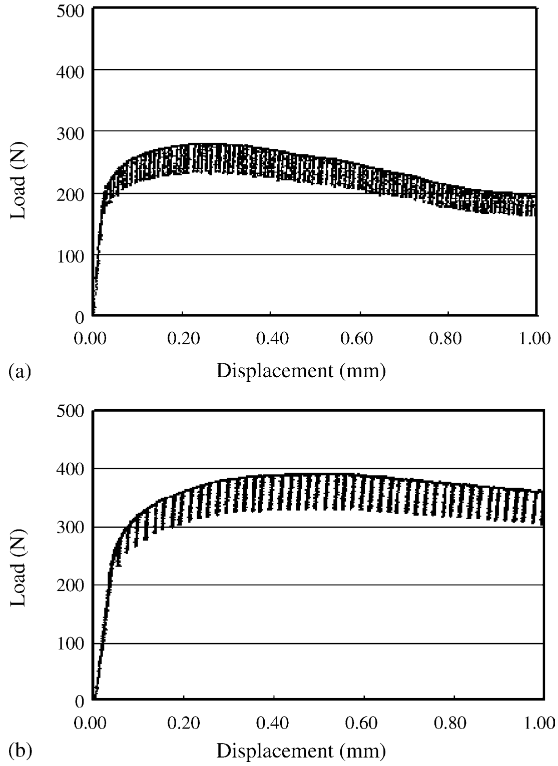

Fig. 3. Load and displacement curves obtained from: (a) DFMB and (b) pre-cracked t/2-1/3CVN specimens of F82H-std (low N).

3.2. Fracture toughness testing of 0.18DCT

Fig. 4 shows the DCT test machine, which is manufactured to obtain fracture behavior for the 0.18DCT specimen. Fig. 4(c) shows the outboard gage attached to one of the disk compact specimen of F82H-std steel. This fracture toughness tests can be conducted in the temperature range from −180 to 300 °C, and the temperature was controlled by the system of LN_2 vapor or electric heater. Tensile force, clip gauge displacement, cross head displacement and temperatures are measured and recorded during the tests. The load versus

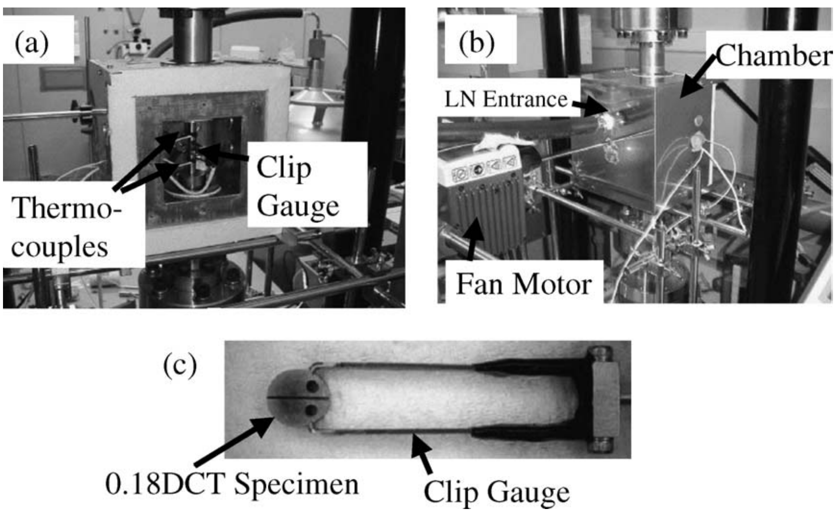

Fig. 4. 0.18DCT machine: (a) chamber inside, (b) front of chamber and (c) clip gauge and specimen.

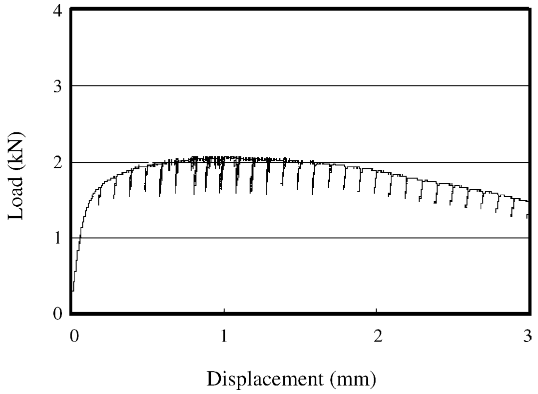

Fig. 5. Load and displacement curves obtained from: (a) DFMB specimen and (b) t/2-1/3PCCVN of F82H-std (low N).

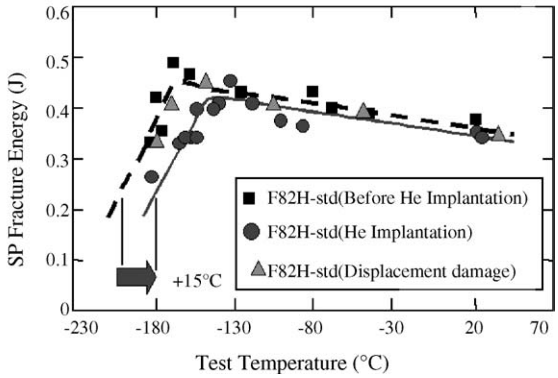

Fig. 6. SP energy as a function of temperature in F82H-std (IEA).

clip gage displacement curves are shown in Fig. 5. The displacement rate of the cross head was controlled at 0.2 mm/min under the unloading compliance method. Fracture toughness of F82H-std (low N) at RT was about 330 MPa $m^{1/2}$. This value is very similar to the value of previous study [9].

4. Small punch testing

Small punch (SP) test machine was manufactured in a hot cell of the JMTR hot laboratory [10]. The SP test machine consists of a load controller, turntable with 12 specimen holders, a vacuum chamber and a furnace. The specimen holder consists of the upper and lower holders, a punch and a steel ball of 1 mm diameter. A steel ball and a punch were pushed by the punch rod. The maximum load and stroke of the punch rod for the SP machine are 5 kN and 8 mm, respectively. The punch speed was controlled at 0.5 mm/min. SP energy was calculated from the area under the load–deflection curve up to the fracture load.

In this study, the F82H specimens of TEM disk type with 0.3 mm thickness were irradiated through F82H foil of 0.6 mm at about 120 °C with a beam of 100 MeV He^{2+} particles by AVF cyclotron at Takasaki Ion Accelerators for Advanced Radiation Application (TIARA) facility of JAERI. The displacement damage was about 0.03 dpa and the stopping range of helium was about 1.25 mm. In this specimen, all helium atoms passed through the irradiated specimens. After the irradiation, the SP tests were performed in the Japan Materials Testing Reactor (JMTR) Hot Laboratory. The SP energy was calculated as a function of temperature as shown in Fig. 6. In this study, the change of DBTT due to displacement damage hardly occurred in this experiment and it was about 2–3 °C. In the previous study [11] of 50 MeV He^{2+} irradiation experiment, displacement damage in F82H steel was also about 0.03 dpa and the projected range of the helium ions controlled under an energy degrader was from 0 to 0.4 mm, and helium atoms were uniformly implanted to about 85 appm at about 100 °C in the specimen with 0.3 mm in thickness. The shift of DBTT for the F82H steel implanted with 85 appm He was about 15 °C [11]. The summaries of SP data tested at RT in F82H steel were given in Table 2. In the similar cyclotron helium implantation experiment of Kimura,

Table 2
SP-yield load, SP-maximum load, cracking load, deflection at a maximum of load and total elongation of F82H steels tested at RT by SP

	Displacement damage (dpa)	He (appm)	Yield load (N)	Maximum load (N)	Cracking load (N)	Deflection at P_{max} (mm)	Total deflection (mm)
F82H	0	0	123	522	348	0.38	0.78
F82H (50 MeV He)	0.03	85	120	493	337	0.38	0.75
F82H (100 MeV He)	0.03	0	123	499	350	0.48	0.74

it was reported that the shift of DBTT due to helium implantation of 120 appm was about 20 °C in JLM-1 steel [12]. In our previous data, the ratio of the shift of DBTT to helium concentration in F82H steel was about 0.18 °C/appm He, and the ratio for Kimura data in JLM-1 steel was 0.22 °C/appm He. These two data for the helium effect on DBTT in different martensitic steels were very similar. The DBTT obtained by the our previous SP experiments in F82H steel could be modified as 37.5 °C for the DBTT measured using a 1/3CVN standard as determined from the correlation between SP data and 1/3CVN data [13]. The shift of DBTT due to displacement damage at 0.03 dpa can be evaluated from the other data of neutron irradiation experiment [14] and the value is estimated as about 5 °C. In this experiment of F82H steel, the DBTT shift due to displacement damage of 0.03 dpa can be evaluated as about 6 °C, and the value obtained by this study is very close to the result of the other study. Therefore, the shift of DBTT due to helium production of 85 appm could be concluded to be about 32 °C in the 1/3CVN. In present study, the same result was obtained. On the other hands, in boron or nickel doping experiments of martensitic steels, similar results of the shift of DBTT due to helium production on the isotope-tailoring experiments were reported [14,15]. However, there was a possibility that the addition of boron or nickel may cause severe irradiation embrittlement. In this experiment, there are no effects of chemical additional element on DBTT [16,17], the shift of DBTT [11], the increment of irradiation hardening [18,19] and defect clusters [20,21] as reported in previous studies. It is therefore concluded that helium production can affect the shift of DBTT.

5. Charpy impact tests

Fig. 7 shows the dependence of Charpy impact energy on specimen size as a function of temperature, using t/2-CVN, 1/3CVN and t/2-1/3CVN specimens of F82H-std containing 20 ppm N (low N). The upper shelf energy per cross-section in fracture plane was decreasing with the reducing specimen size. The DBTT of t/2-CVN, 1/3CVN and t/2-1/3CVN was −82, −104 and −140 °C, respectively. It is well known that DBTT depends strongly on the width of specimen (B), and the length of ligament below the notch of the specimen (b)

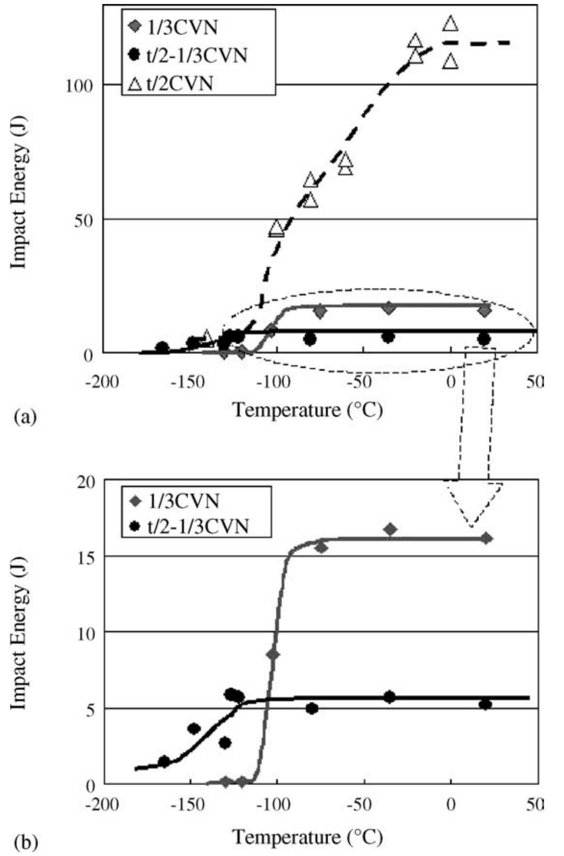

Fig. 7. Dependence of specimen size on Charpy impact energy as a function of temperature, using t/2-CVN, 1/3CVN and t/2-1/3CVN specimens of F82H-std (low N).

[22–25]. The empirical correlations of DBTT of full-size and sub-size specimens in reactor pressure vessel (RPV) steels were proposed:

$$\mathrm{DBTT}_{\text{full-size}} = \mathrm{DBTT}_{\text{sub-size}} + 98 - 15.1 \times \ln(Bb^2), \tag{1}$$

where $\mathrm{DBTT}_{\text{full-size}}$ and $\mathrm{DBTT}_{\text{sub-size}}$ are transition temperature for full-size and sub-size Charpy impact specimens, respectively [22]. The values of DBTT, B and b in this study are given in Table 3, and the DBTT for 1/3CVN and t/2-1/3CVN can be estimated as about −130 and −141 °C, respectively, by using Eq. (1). The estimated value of DBTT for t/2-1/3CVN was good corresponds to the experimental data of F82H steel in this study, but the estimated value of DBTT for 1/3CVN was lower than the value of the present experimental

Table 3
DBTT, width of specimen (B) and the length of ligament below the notch (b), of F82H steel for t/2-CVN, 1/3CVN and t/2-1/3CVN Charpy impact specimens

	Full-size or sub-size	B (mm)	b (mm)	DBTT (°C) (experimental data)	DBTT (°C) (estimation)
t/2-CVN	Sub-size	5	8	−82	(−82)
1/3CVN	Sub-size	3.3	2.79	−104	−130
t/2-1/3CVN	Sub-size	1.65	2.79	−140	−141
CVN	Full-size	10	8	–	−81.6

The estimation of DBTT in t/2-CVN, 1/3CVN and CVN was calculated by using a data of t/2-CVN.

data. The DBTT of F82H steel was lower than that of RPV steel, and the other factors such as the size and density of inclusions may be related to the correlations of DBTT for the size dependence. Further study is needed for the empirical correlations of DBTT of full-size and sub-size specimens in RAF steels.

6. Summary

(1) New bend test machines have been developed to obtain fracture behaviors of F82H steel for very small bend specimens of t/2-1/3PCCVN with 20 mm length and DFMB with 9 mm length and disk compact tension of 0.18DCT type, and fracture behaviors were examined at 20 °C.
(2) The effect of specimen size on DBTT of F82H steel was examined by using t/2-CVN, 1/3CVN and t/2-1/3CVN, and it was revealed that DBTT of t/2-1/3CVN and 1/3CVN was lower than that of t/2-CVN.
(3) DBTT shift due to helium production and displacement damage was examined by small punch tests.

Acknowledgements

The authors would like to express sincere thanks to Drs. M. Ando and T. Sawai of JAERI for their helpful discussions and the staffs of hot laboratory of JMTR and JMTR operation in Oarai establishment of JAERI.

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