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Mechanical Properties of Neutron Irradiated F82H using Micro-Tensile Testing

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Abstract

Room temperature micro-tensile tests were successfully performed on F82H specimens neutron-irradiated at 573 K up to 5 dpa and unirradiated using a focused ion beam (FIB) device. Dimensions of the gauge section for micro-tensile specimens were about 10 μm (length) x 1 μm^2 (area). These specimens were fabricated in a FIB device. The tensile properties obtained on specimens of micrometer size qualitatively agreed with results from millimeter size specimens. Using this micro-tensile testing technique is considered to become very useful data in combining the micro-tensile data on ion irradiated F82H with data on neutron irradiated material. This method overcomes the limitation on obtaining mechanical property on ion irradiated specimens to only micro-indentation testing.

Key words: ultrasmall testing technology, micro-tensile testing, neutron irradiation, reduced activation Ferritic/Martensitic steel

1. Introduction

Reduced activation ferritic/martensitic steels (RAFM) are the most promising candidates for blanket structural materials of DEMO fusion reactors [1]. As well known, neutron irradiation of RAFM causes remarkable changes in mechanical properties, especially below irradiation temperatures of 573K [2]. Furthermore, some transmutations (mainly helium atoms) are produced by 14 MeV neutrons on the first wall. Mechanical property data can be obtained by neutron irradiation experiments in fission reactors. However, only low concentrations of helium atoms are produced in F82H by neutron irradiation in a fission reactor. Therefore, it is very difficult to evaluate any helium effects on mechanical properties. On the other hand, ion irradiation is a convenient and accurate way to simulate the 14 MeV neutron irradiation condition because damage rates and helium co-implantation rates of specimens can be controlled by using MeV-class dual-ion accelerators. Unfortunately, the ion irradiated layer is limited to the small volume, near surface layers. Recently, the irradiation hardening behavior of ion bombarded F82H is becoming clear from the results of micro-indentation hardness testing. However, it is difficult to obtain other mechanical properties (e.g., deformability) from these results. Thus, an evaluation of mechanical properties using the ultra-small testing technologies (USTTs) is also being started by using micro-pillar and micro-tensile testing methods with ion irradiated F82H. This work is done as part of the Broader Approach (BA) collaboration activities [3,4]. Currently, it has not have been clear how to correlate measurements between the millimeter size and micrometer size specimen deformation behavior for the irradiated F82H. The effects of specimen size can be evaluated using the neutron irradiated F82H millimeter size specimens irradiated in a fission reactor. The technique can then be used to evaluate micro-tensile results from ion-irradiated F82H, and compare the micro-tensile results with tensile properties of millimeter size specimens.

The purpose of this study is to investigate the mechanical properties of F82H steels after neutron irradiation at 573 K up to 5 dpa by using micro-tensile testing and compare with the tensile properties obtained on SS-3 type tensile specimens.

2. Experimental procedures

The materials used are reduced activation ferritic/martensitic steel F82H IEA heat (Fe-8Cr-2W-VTa) [5] and pure iron [6]. The small pieces of neutron irradiated F82H steel which were irradiated at 573 K, 5 dpa (nominal condition) in HFIR were transported to Japan after the post irradiation experiment (Charpy impact test) in a hot laboratory in Oak Ridge National Laboratory. Micro-tensile tests were conducted on this material (ID:A006 [7]). In this study, a micro-tensile specimen was fabricated by a focused ion beam (FIB) device (Hitachi FB-2100A with a micro-sampling system). The specimen was a sheet type with the gauge section of ~10x1x1 μm as shown in Fig. 1a). However, the actual size of gauge section was measured by SIM image in FIB during fabrication of each specimen. And then the engineering stress was calculated from the actual size. The micro-tensile testing procedure is shown in Fig. 1b), which is basically similar to the preparation of TEM thin foils. Micro-tensile testing was performed by using a tungsten needle micro-sampling system in the FIB micro-process chamber [8]. The lower side of the specimen is fixed to a micro-beam (CVD-SiC small bar) having a size of 4x7x100 μm in which a load-displacement curve was obtained. The upper side is fixed to a tungsten needle by tungsten deposition. The tensile test was performed by an additional manipulator controller that can move this tungsten needle only in the direction of the tension axis. The tungsten needle can be move for a constant movement (about 0.18 μm/15steps) without a tensile specimen by this controller. The actual load during micro-tensile testing was estimated from the deflection of the micro-beam at that time in the FIB secondary ion microscopy (SIM) image. For the estimation of the strain, markers were produced at both ends of the gauge section by tungsten deposition, and the strain was calculated from the distance between the markers. The micro-tensile test was carried out in the FIB device, for tests at room temperature under vacuum. Microstructural observation was performed on the fractured specimen after the micro-tensile test. First, the specimen was fixed to the surface of another F82H steel section by tungsten deposition, and then a thin foil was fabricated by the FIB device. The microstructural examination was carried out using a JEOL JEM-2100F transmission electron microscope (TEM) operating at 200 kV.

3. Results and discussion

3-1. Results of micro-tensile testing of unirradiated / irradiated F82H

Figures 2a)-c) show examples of SIM images for the micro-tensile testing of a) unirradiated F82H, b) pure-iron and c) neutron irradiated F82H. The tungsten needle was fixed to an upper side of the specimen by tungsten deposition and then moved to perform the tensile testing. For unirradiated F82H as summarized in Fig. 2a), a uniform elongation (ε_u) occurred even though it was extremely small up to 100 steps. Local deformation due to shear slip occurred within the gauge section and then the specimen broke without necking. Pure iron was also investigated to compare with the deformation behavior of F82H as shown Fig. 2b). These tensile specimens were fabricated without grain boundaries in the gauge section. Unlike F82H steel, pure iron tends undergo a large strain up to the ultimate tensile strength (UTS). In the SIM images, some distortion which seems to be deformation in the slip direction was observed in the gauge section of specimen. Finally, for neutron irradiated F82H in Fig. 2c), the number of steps until fracture is obviously small as compared with results of the unirradiated F82H specimen. Once local slip deformation occurs in the gauge section the fracture occurs quickly with little or no additional deformation.

Figure 3 shows a cross-sectional TEM observation for an upper side half specimen of unirradiated F82H after micro-tensile test. Since the F82H steel is tempered martensite, the dislocation density is very high in an unirradiated specimen. However, a higher dislocation density microstructure was observed at the tip of the fractured specimen. The deformation is not spread over the entire gauge section of the specimen but tends to concentrate locally. On the other hand, for neutron-irradiated F82H, results of deformed microstructure after tensile testing of SS-3 type specimen irradiated at 573K up to 5 dpa were reported [9]. The deformed microstructure will be also observed for broken specimens tested by this study, and we will compare with characterization of these SS-3 specimen deformed microstructures in the future.

3-2. Comparison of tensile properties of millimeter size and micrometer size specimens

Figure 4b) shows the results of engineering stress-strain curves which were estimated from SIM images in Figs. 2a) and 2c). For unirradiated F82H, the yield stress (YS) was 457 MPa, the total elongation (ε_t) was about 10%. For neutron-irradiated F82H, yield stress and ultimate tensile strength levels were almost equal at 635 and 636 MPa, respectively. These stress levels become almost same because the work hardening ability of irradiated F82H is reduced due to irradiation hardening and the uniform elongation is near zero. The total elongation of the irradiated material was less than 5%. Figure 4a) shows the engineering stress-strain curves which were obtained on SS-3 tensile specimens of unirradiated and neutron-irradiated F82H [10]. Table 1 summarizes tensile properties obtained from micro-tensile testing, and tensile properties data (ID: A026 [11]) for SS-3 type specimens (millimeter size), included to compare results from the two specimen sizes. (This is a different specimen than the one used for the small piece from which micro-tensile specimen was fabricated. However, the irradiation condition is very similar.) Therefore, it was considered that these tensile properties can be compared in this study. Furthermore, in recent study [12], this strain is also being obtained by a distance change of markers on the specimen during the tensile test with

NCDMS (Non-contact deformation measurement system). A measurement of displacement for irradiated tensile specimens as SS-J3 can be performed more accurately by NCDMS. Therefore, the deformation behavior of tensile tests will be evaluated by this method in the future.

Qualitatively, the yield stress and ultimate tensile strength of the irradiated F82H at 573 K up to 5 dpa increase both for millimeter and micrometer size specimens. Additionally, the tendency of a decrease in uniform and total elongation is consistent. Basically, it is reported that the plate like small tensile specimens in RPV steel have the effect of specimen geometry. For example, they are thickness-to-width (T/W) and length-to-width (L/W) ratio as scale factor. When the thickness of small tensile specimen is less than 0.2mm, the deformation (especially, total elongation) become to decrease [13]. Furthermore, Gussev, et al. performed the tensile test using sub-size tensile specimens (SS-Mini) [14]. The tendency of their tensile properties is similar to results in this study although micro-tensile specimens is smaller size.

However, quantitatively,

・ The yield stress and ultimate tensile strength tend to be lower in the micrometer size specimen, for both unirradiated and irradiated material.

・ The total elongation of the micrometer size specimen is smaller than that of a millimeter size specimen in both unirradiated and irradiated F82H.

This behavior is caused by the difference in the evaluation size for each test method. Figure 5 shows that relationship between the evaluation size of each test method and the size of microstructural features in a tempered martensite like F82H. It is well known that tempered martensitic steel has the characteristic martensitic structures and also has high strength and resistance to fracture. In this case, strength of a micro-tensile test shows a lower level because micro-tensile specimens only including part of the martensitic structural features. It shows that strengthening of boundaries decreased due to the small volume of the specimen. For example, Ohmura et al., reported that the amount of block boundaries of martensite affected the strength of Fe-C alloys [15].

Also, elongation of the millimeter size specimen showed a larger deformation. The reason is that the deformation is not highly concentrated in a single grain immediately after yield stress in a millimeter size specimen but can be in a micrometer specimen. Probably, the deformation in the millimeter specimen also spreads to other grains/boundaries. From result in Fig. 3, the microscopic deformation behavior (including several boundaries) can be investigated by the micro-tensile test method. However it is also necessary to consider the possibility of different deformation mechanisms in martensite steel with the many boundaries and structures of a macro-size specimen. It is reported that very high density small defects were formed in the lath matrix in neutron-irradiated F82H at 573 K [9]. These defects become the barriers to slip dislocations which contribute to tensile deformation. Consequently, the deformation is not easily spread during tensile testing, and. tensile elongation of neutron-irradiated F82H decreases in both millimeter and micrometer size specimens. Additionally, it should be considered that the scatter of micro-tensile data is very large for an evaluation using these microscopic volume specimens. It is possible to obtain tensile properties of a micrometer volume including some lath boundaries of F82H by using micro-tensile specimens although it is necessary to have more data points from micro-tensile testing. Based on these results, future work will clarify the effects of helium and hydrogen on tensile properties for F82H by using co-implantation with helium and hydrogen.

4. Conclusion

This study examined the mechanical properties of neutron-irradiated F82H using a micro-tensile test method;

1) Micro-tensile tests were successfully performed for unirradiated and neutron-irradiated F82H specimens at room temperature.

2) The change in tensile properties due to neutron irradiation agreed qualitatively for both micrometer and millimeter size F82H specimens.
3) A high dislocation density microstructure was observed at the tip of a fractured specimen of unirradiated F82H. The deformation tends to concentrate locally in the micro-tensile specimen of F82H.
4) The results of micro-tensile testing of neutron-irradiated F82H will be very useful in combining the micro-tensile data from ion-irradiated F82H with neutron irradiated data. This expands the types of mechanical property test data that can be obtained from ion-irradiated specimens beyond that available from micro-indentation test.

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[1] H. Tanigawa, K. Shiba, A. Moslang, et al., J. Nucl. Mater. 417 (2011) 9-15.

[2] H. Tanigawa, E. Gaganidze, T. Hirose, et al., Nucl. Fusion 57 (2017) 092004.

[3] R. Kasada, S. Konishi, K. Yabuuchi, et al., Fusion Eng. Des. 89 (2014) 1637-1641.

[4] T. Miura, K. Fujii, K. Fukuya, et al., submitted to this proceeding.

[5] K. Shiba, et al., 1997 JAERI-Tech 97-038 (Japan Atomic Energy Research Institute, 1997).

[6] E. Wakai, JAEA-Research 2006-012 (Japan Atomic Energy Agency, 2006) 7.

[7] K. E. Lenox and M. L. Grossbeck, Fusion Reactor Materials, DOE/ER-0313/25 (1998) 310.

[8] T. Miura, et al., J. Nucl. Mater. 457 (2015) 279-290.

[9] N. Hashimoto, et al., Fusion Sci. and Technol. 44 (2003) 490-494.

[10] K. Shiba, et al., Fusion Reactor Materials, DOE/ER-0313/28 (2000) 131-135.

[11] K. E. Lenox and M. L. Grossbeck, Fusion Reactor Materials, DOE/ER-0313/25 (1998) 313.

[12] H. Sakasegawa, X. Chen, T. Kato et al., submitted to this proceeding.

[13] T. S. Byun, et al., ASTM STP 1329 (1998) 575-587.

[14] M. Gussev, et al., ASTM STP 1576 (2014) 1-19.

[15] T. Ohmura, et al., J. Mater. Res. 18 (2003) 1465-1470.

Figure captions

Figure 1 Detail of micro-tensile testing method.

a) Dimension of micro-tensile specimen (sheet type specimen)

b) Experimental procedure for micro-tensile testing

Figure 2 Secondary ion microscopy (SIM) images under micro-tensile testing in FIB device.

a) F82H IEA (Unirradiated), b) Pure-iron (Unirradiated)

c) Neutron irradiated F82H IEA (5dpa, 573K)

Figure 3 A Cross-sectional TEM images of microstructure for unirradiated F82H after micro-tensile testing.

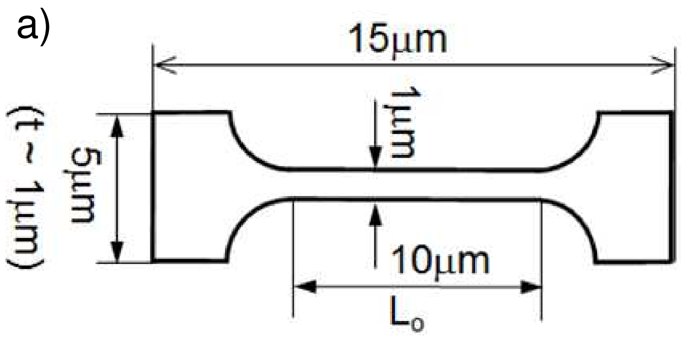
Figure 4 Stress-strain curves of millimeter and micrometer F82H specimens.

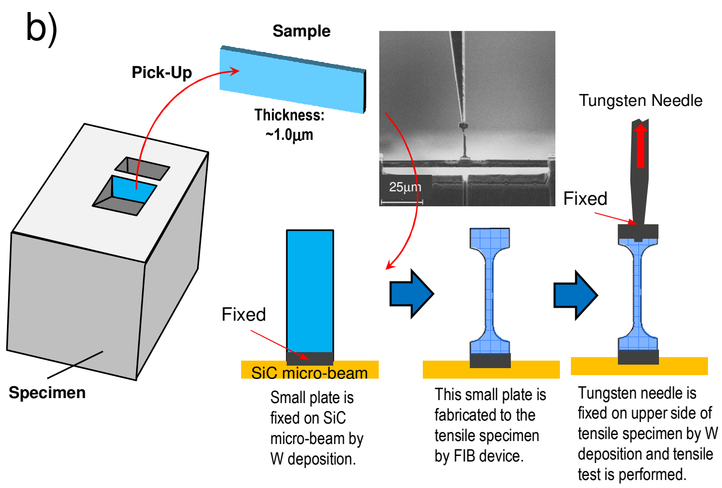
a) obtained with SS-3 specimens for unirradiated / neutron-irradiated F82H IEA. (Strain rate: $1x10^{-3}$ /s).

b) obtained with micro-tensile specimens.

Figure 5 Comparison of evaluated areas for each test method.

Table 1 Summary of tensile properties of F82H obtained using millimeter and micrometer specimens.





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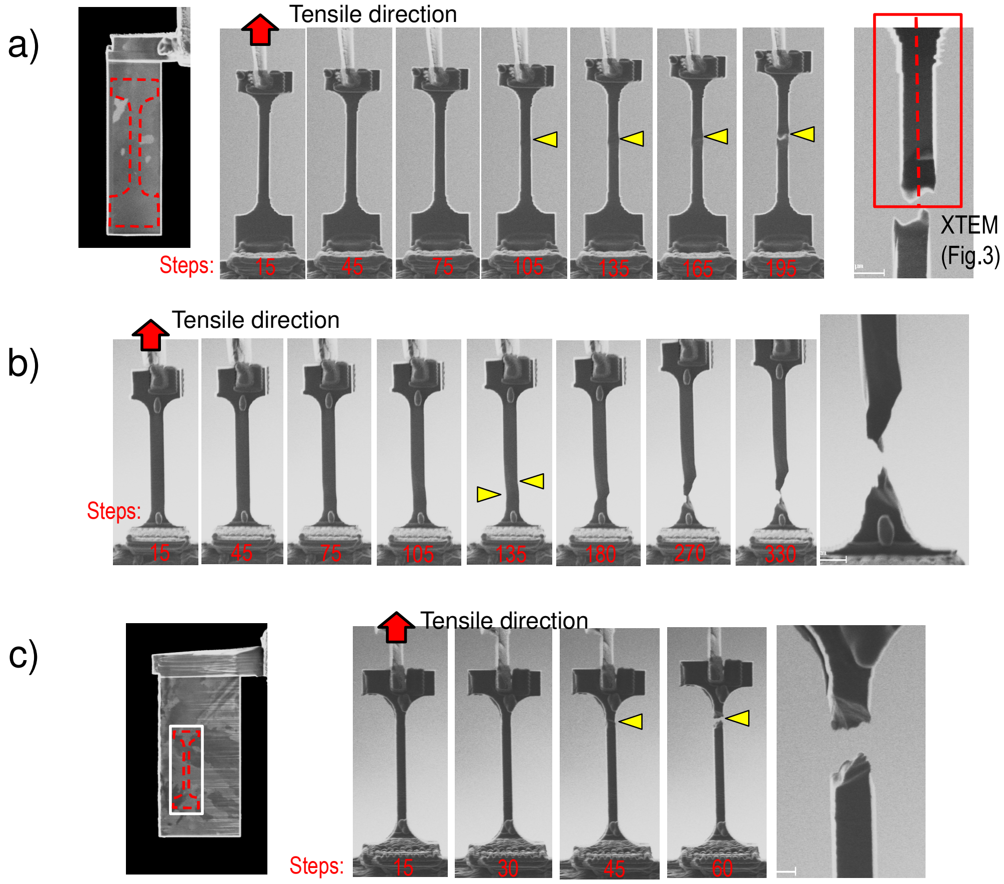

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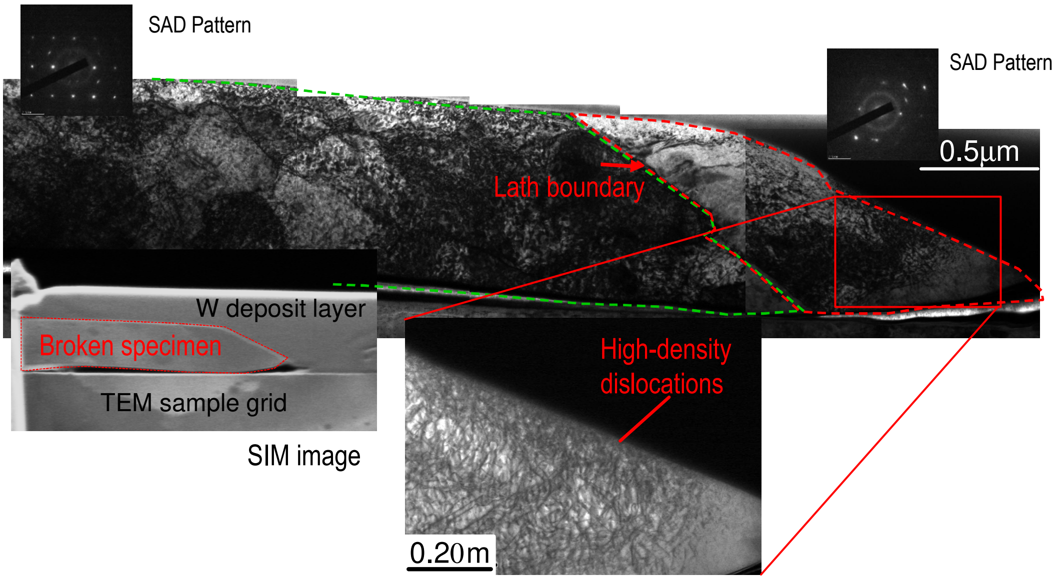

Figure 3 A Cross-sectional TEM images of microstructure for unirradiated F82H after micro-tensile testing.

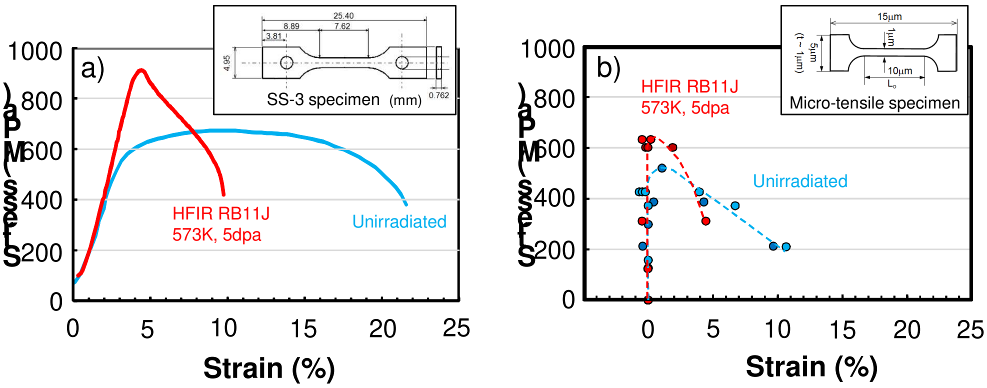

Figure 4 Stress-strain curves of millimeter and micrometer F82H specimens.

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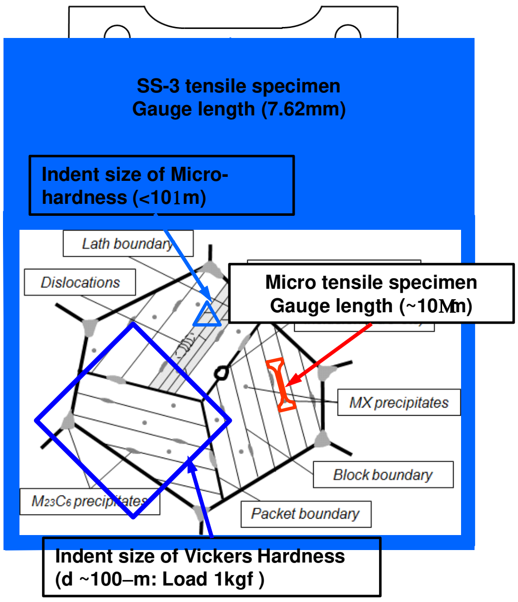

Test method versus evaluation size

Test Method	Micro-Indentation	Micro-Tensile	Vickers Hardness	Tensile Test
	△		◇	
Scale	~10Mm	~10Mm	~100µm	~mm
Micro structure	Lath	Lath /Boundary	Lath Block Packet Precipitates	ALL
Irradiation	Ion or Neutron	Ion or Neutron	Neutron	Neutron

Figure 5　Comparison of evaluated areas for each test method.

Table 1 Summary of tensile properties of F82H obtained using millimeter and micrometer specimens.

	SS-3 tensile results (mm-size) A023 (RB11J 4.9dpa, 580K)				Micro-tensile results (μm-size) A006 (RB11J 4.9dpa, 567K)			
Tensile properties	YS (MPa)	UTS (MPa)	Υ_u (%)	δ_t (%)	YS (MPa)	UTS (MPa)	τ_u (%)	T_t (%)
Neutron irrad. F82H	898	911	0.45	7.8	635	636	0.21	4.4
Un-irrad. F82H	534	651	6.3	19.5	457	519	1.1	10.2
Irrad. Hardening (53)	364	260	–	–	178	117	–	–

Dose rate was about 2×10^{-7} dpa/s.

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Highlights

・Room temperature micro-tensile tests were successfully performed on unirradiated and neutron irradiated F82H.

・The engineering stress-strain curves were obtained by micro-tensile testing of neutron irradiated F82H.

・The tensile properties of micrometer and millimeter size specimens were roughly in qualitative agreement.