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**Effect of Initial Heat Treatment on Tensile Properties and
Charpy Impact Properties of Reduced-Activation
Ferritic Steel F82H Irradiated by Neutrons**

Eiichi WAKAI

Research Group for Irradiation Field Materials
Nuclear Science and Engineering Directorate

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Effect of Initial Heat Treatment on Tensile Properties and Charpy Impact Properties of Reduced-Activation Ferritic Steel F82H Irradiated by Neutrons

Eiichi WAKAI

Division of Fuels and Materials Engineering

Nuclear Science and Engineering Directorate

Japan Atomic Energy Agency

Tokai-mura, Naka-gun, Ibaraki-ken

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Effects of initial heat treatments on irradiation hardening and embrittlement of reduced-activation martensitic F82H steel were mainly examined by the experiments of neutron irradiations. From the analysis of the changes of yield stress and ductile-brittle transition temperature (DBTT) due to irradiation, it was found that the controll of heat treatment of tempering before irradiation was very useful for the improvement of irradiation hardening and embrittlement.

Keywords: Neutron Irradiation, Yield Stress, Ductile-Brittle Transition Temperature, DBTT, F82H, Reduced-Activation Ferritic/Martensitic Steels, Heat Treatment, Irradiation hardening, Irradiation embrittlement

中性子照射した低放射化フェライト鋼 F82H の引張特性とシャルピー衝撃特性に及ぼす

照射前の熱処理効果

日本原子力研究開発機構 原子力基礎工学研究部門 燃料・材料工学ユニット

若井 栄一

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核融合炉構造材料として開発している F82H 鋼の照射硬化と照射脆化に及ぼす照射前の熱処理効果について、JMTR 等の原子炉で中性子照射実験を行って検討した。照射前後での延性脆性遷移温度 (DBTT) と降伏応力の変化を解析した結果、照射後の強度特性は照射前に行う焼き戻し時間や温度の調整によって、その性能を向上させることができた。

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1. Effect of Tempering Temperature and Time on Tensile Properties of F82H Irradiated by Neutrons

1.1 Research Background and the Purpose of This Study

Reduced-activation ferritic/martensitic steels are candidate materials for the blanket structure of fusion reactors. Radiation-hardening of 9%Cr martensitic steels irradiated by neutrons occurs mainly at irradiation temperatures lower than about 400°C, and it increases with decreasing irradiation temperature down to 250°C[1,2]. The shift of DBTT also increases with decreasing irradiation temperature, and the shift increases largely for irradiation at 250°C[3,4]. Several researchers [1-7] reported that the increase of yield strength and the shift of DBTT were different in several martensitic steels, such as F82H, JLF-1, JLF-1B, ORNL 9Cr-2WVTa, OPTIFER Ia, II, MANET II and Mod.9Cr-1Mo, which had different concentrations in some elements and were tempered at different temperatures. The effects of the normalizing and tempering of heat treatment on tensile and impact behavior in martensitic steels before irradiation were reported by L. Schafer [8] and P. Gondi [9]. However, the mechanisms of the changes of yield strength and DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment and impurities on them. The optimum heat treatment will be required to improve resistances to radiation hardening and embrittlement. In this study, the dependence of tensile behavior on tempering time and temperature has been examined for martensitic steel F82H (Fe-8Cr-2W-0.2V-0.04Ta-0.1C) irradiated at 250°C to a neutron dose of 1.9 dpa.

1.2 Experimental Procedure

The chemical compositions of F82H, F82H+2Ni, Fe+0.1C and pure iron used in this study are shown in Table 1.1. The specimens were first normalized at 1040°C for 0.5 h and tempered at 750°C for 1 h. A second heat treatment was performed on the F82H steel, which was secondly normalized at 1040°C for 0.5 h and tempered at temperatures of 750, 780 and 800°C for 0.5 h. The tempering time at 750°C was varied between 0.5 and 10 h. SS-3 tensile specimens were prepared from the normalized and tempered F82H, F82H+2Ni, Fe+0.1C and pure iron. The SS-3 sheet tensile specimens were 0.76 mm thick with a gage length of 7.6 mm. Irradiation was carried out in the Japan Materials Test Reactor (JMTR) to neutron fluences of $1.2 \times 10^{21} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) and $2.2 \times 10^{21} \text{ n/cm}^2$ ($E < 0.5 \text{ MeV}$), resulting in a displacement damage value of 1.9 dpa. Capsule 00M-61A, nominally at 250°C, was a shrouded type with reactor coolant flowing over an aluminum cladding tube containing the specimens; gamma-heating was used to raise the specimen temperature. After irradiation, tensile testing was carried out in air at room temperature at a strain rate of $4.4 \times 10^{-4}/\text{s}$.

1.3 Tensile Properties of F82H Steels irradiated at 250°C

The changes of tensile properties with tempering temperature and time in F82H steel irradiated at 250°C to 1.9 dpa are also shown in Figs. 1.1 and 1.2. The summary of tensile data of the irradiated and non-irradiated specimens is given in Table 1.2. The yield stress and the increment of yield stress caused by irradiation, radiation hardening, in F82H tempered at 750, 780 and 800°C for 0.5 h are shown in Fig. 1.1(a) and 1.1(b), respectively. The yield stress of F82H steel tempered at 780 and 800°C was lower than that tempered at 750°C before irradiation, but the former was slightly higher than the latter after irradiation. As a result, the radiation hardening of F82H steel tempered at 780 and 800°C was about twice as large as that of F82H steel tempered at 750°C. Radiation hardening of F82H steel heated by the first normalizing- and -tempering (N&T) treatment was considerably larger than that by the second N&T treatment. The loss of ductility due to irradiation was recovered by the second N&T treatment from 5.4 to 3.6 %. In Fig. 1.2, the dependence of yield stress on time of tempering in F82H steel tempered at 750°C was given, and the increment of yield stress largely changed between 1 and 2 h for the time of tempering. The change of yield stress of the irradiated pure iron, Fe+0.1C, F82H and F82H+2Ni is given in Fig. 1.3. The radiation hardening of F82H steel was comparable to that of pure iron and Fe-0.1C alloy, but the radiation hardening of F82H+2Ni increased. The loss of ductility of pure iron and Fe+0.1C largely occurred. The ability to strain harden was completely lost for pure iron (UE ~ 0.1%) and was very low for F82H (UE: from 0.1 ~ 0.5%) and relatively high for Fe+0.1C (UE ~ 1.2%) and F82H+2Ni (UE ~ 8.3%).

Microstructures of martensitic steels, such as dislocations, carbides and lath width, depend strongly on the temperature and time of tempering. The density of dislocations of F82H decreased with increasing temperature and increasing time of tempering [10]. The concentration of carbon in solution in the matrix will increase with tempering temperature and time. In the specimen tempered at higher temperatures, the sink density for point defects due to dislocations becomes lower and the formation of dislocation loops may increase and the growth rate of dislocation loops will increase. The mobility of interstitial atoms in martensitic steels will be reduced with increasing an concentration of carbon in solution, and the formation of dislocation loops will also increase. Radiation-hardening can be evaluated by Orowan's theory for athermal bowing of dislocations around obstacles on a slip plane [11-13] and depends on the number density and size of defect clusters. The changes of sink density and the concentration of carbon in solution can affect the formation and growth of dislocation loops, and therefore, radiation hardening should depend on the temperature and time of tempering.

As shown in Figs. 1.1 and 1.2, the radiation hardening of F82H was reduced by the second N&T treatment, however, the reason for the difference in radiation hardening between the first N&T and second N&T can not be exactly explained by that alone, and it needs further investigation.

The effects of tempering in martensitic steel on radiation hardening were also investigated by a micro-indentation technique using specimens irradiated with high-energy ions up to high dose levels [14]. The initial microstructures related to tempering conditions also affected the swelling behavior [10,15,16], and therefore the control of tempering conditions is a very important to provide the steels high resistances

to radiation hardening, radiation embrittlement and swelling.

1.4 Summary

The dependence of tensile properties on tempering time and temperature was examined for a martensitic steel F82H irradiated at 250°C to a neutron dose of 1.9 dpa in the Japan Materials Testing Reactor. The specimens were first normalized at 1040°C for 0.5 h and tempered at 750°C for 1 h. A second heat treatment was performed at temperatures from 750 to 800°C for 0.5 h after the normalizing at 1040°C for 0.5 h. The second tempering time at 750°C was varied from 0.5 to 10 h. The tensile specimens of F82H+2Ni, pure iron and Fe+0.1C were also irradiated. Tensile tests were carried out at room temperature. The radiation-hardening of F82H was significantly relieved by the second heat treatment, and it was smaller than that of the first heat treated other steels. The radiation-hardening depended on the tempering conditions and tended to increase with increasing the temperature and time of tempering.

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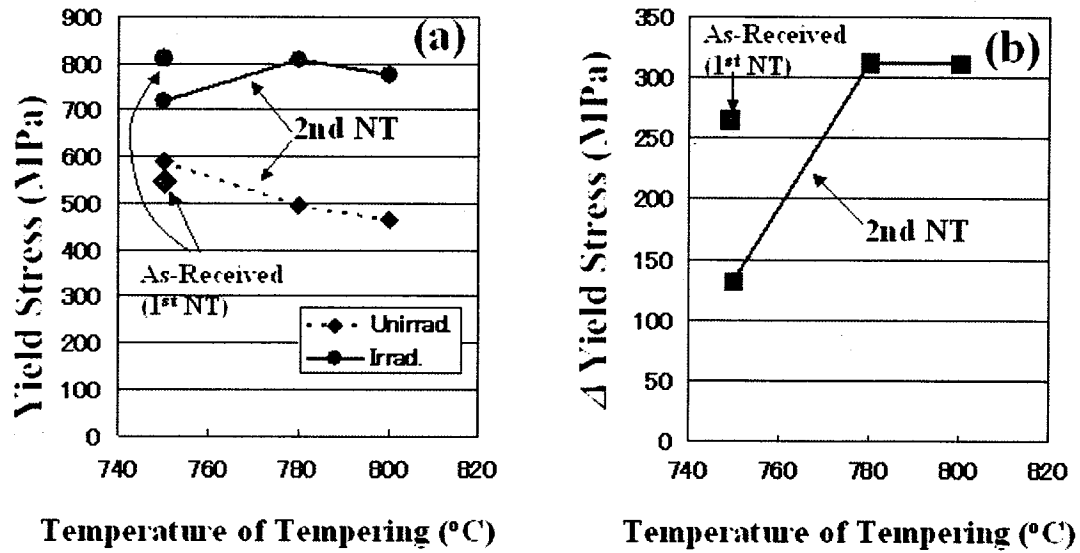


Fig. 1.1 Yield stress (a) and radiation hardening (b) of F82H tempered at 750, 780 and 800 °C for 0.5 h and irradiated at about 250 °C to 1.9 dpa in the JMTR.

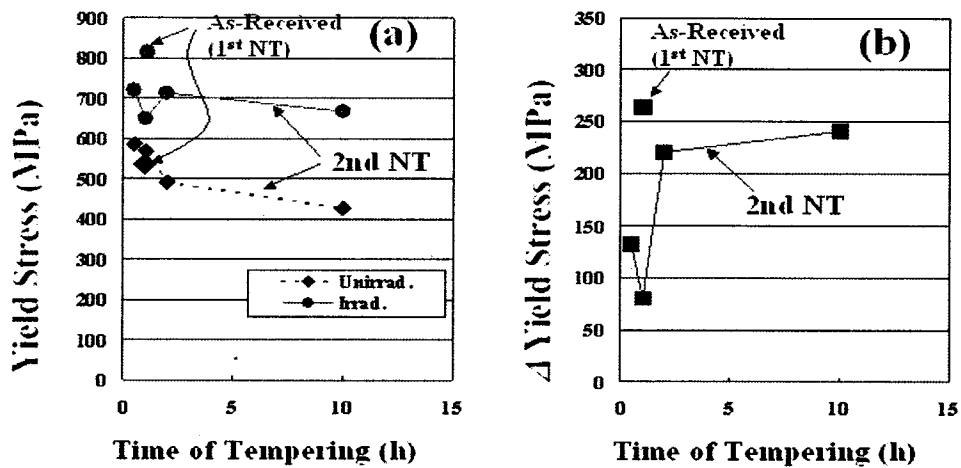


Fig. 1.2 Changes of yield stress (a) and radiation hardening (b) of F82H, tempered at 750 °C for 0.5, 1, 2 and 10 h and irradiated at about 250 °C to 1.9 dpa in the JMTR.

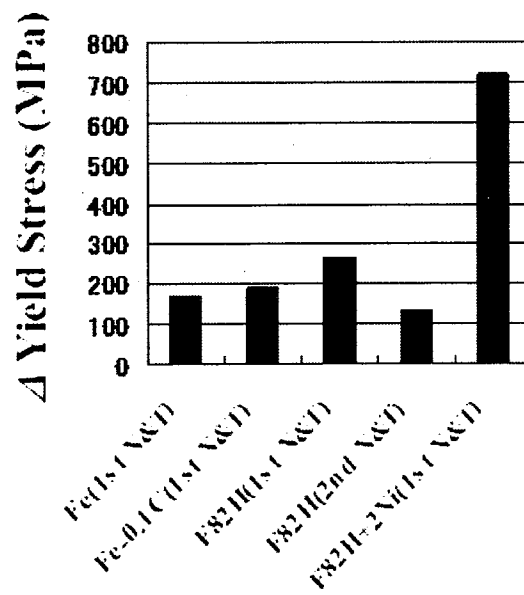


Fig. 1.3 Radiation hardening of pure Fe, Fe-0.1C and F82H+2Ni tempered at 750°C for 1 h and irradiated at about 250°C to 1.9 dpa in the JMTR.

Table 1.1: Chemical compositions of the specimens used in this study (wt%).

Alloy	C	Sol. Al	Si	Mn	P	S	V	Ti	Cr	Ni
F82H	0.09	0.001	0.07	0.1	0.003	0.001	0.19	0.004	7.82	0.02
F82H+2Ni	0.097	<0.001	0.10	0.11	0.003	0.0025	0.19	0.005	7.92	1.97
Fe	0.0019	-	<0.001	<0.0001	0.0003	0.0003	-	-	-	0.0020
Fe-0.1C	0.10	-	<0.001	<0.0001	0.0004	0.0002	-	-	-	<0.0001

Alloy	Cu	Nb	Ta	W	B	O	N
F82H	0.01	0.0002	0.04	1.98	0.0002	-	0.007
F82H+2Ni	-	-	0.06	1.99	-	-	0.0036
Fe	0.0007	-	-	-	0.00002	0.059	0.0002
Fe-0.1C	0.0023	-	-	-	0.00003	0.0015	0.0002

Table 1.2: Summary of tensile properties of F82H, F82H+2Ni, pure iron and Fe-0.1C tested at room temperature. The symbol * indicates the first normalizing and tempering (N&T) heat treatment (The first N&T: 1040°C for 0.5 h and 750°C for 1h). No symbol* shows that the second N&T heat treatment was performed after the first N&T (The second normalizing: 1040°C for 0.5 h).

Alloys	Tempering Temp. (°C)	Tempering Time (h)	Irradiation	Test Temp. (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
F82H	750	0.5	250°C, 1dpa	25	760	786	0.5	12.9
F82H	750	1	250°C, 1dpa	25	708	708	0.4	13.0
F82H	750	2	250°C, 1dpa	25	786	786	0.3	12.3
F82H	750	10	250°C, 1dpa	25	725	725	0.1	11.2
F82H	780	0.5	250°C, 1dpa	25	872	872	0.4	9.7
F82H	800	0.5	250°C, 1dpa	25	846	846	0.1	9.7
F82H	750*	1*	250°C, 1dpa	25	812	812	0.3	10.5
F82H+2Ni	750*	1*	250°C, 1dpa	25	1390	1468	1.2	6.7
Fe	750*	1*	250°C, 1dpa	25	250	250	0.1	11.5
Fe+0.1C	750*	1*	250°C, 1dpa	25	310	363	8.3	19.0
F82H	750	0.5	-	25	588	685	5.0	15.8
F82H	750	1	-	25	569	663	5.5	16.6
F82H	750	2	-	25	492	608	7.0	18.5
F82H	750	10	-	25	428	562	11.0	25.1
F82H	780	0.5	-	25	494	614	7.4	19.6
F82H	800	0.5	-	25	464	592	8.7	21.0
F82H	750*	1*	-	25	548	652	5.5	15.9
F82H+2Ni	750*	1*	-	25	671	802	4.9	14.3
Fe	750*	1*	-	25	83.2	170.2	30.6	48.3
Fe+0.1C	750*	1*	-	25	121	279	31.2	48.0

2. Effect of Initial Heat Treatment on Tensile Properties of F82H Irradiated by Neutrons

2.1 Research Background and The Purpose of This Study

Reduced-activation ferritic/martensitic steels are candidate materials for the blanket structure of fusion reactors and target vessel of spallation neutron source. Radiation-hardening of 9%Cr martensitic steels irradiated by neutrons occurs mainly at irradiation temperatures lower than about 400°C, and it increases with decreasing irradiation temperature down to 250°C [1-2]. The shift of DBTT also increases with decreasing irradiation temperature, and the shift increases largely for irradiation at 250°C [3-4]. Several researchers [1-7] reported that the increase of yield strength and the shift of DBTT were different in several martensitic steels, such as F82H, JLF-1, JLF-1B, ORNL 9Cr-2WVTa, OPTIFER Ia, II, MANET II and Mod.9Cr-1Mo, which had different concentrations in some elements and were tempered at different temperatures. The effects of the normalizing and tempering of heat treatment on tensile and impact behavior in martensitic steels before irradiation were reported by L. Schafer [9] and P. Gondi [9]. However, the mechanisms of the changes of yield strength and DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment and impurities on them. The optimum heat treatment will be required to improve resistances to radiation hardening and embrittlement. In this study, the microstructures of F82H tempered at different conditions has been observed, and tensile properties of F82H, tempered at different conditions, irradiated at 250°C to a neutron dose of 1.9 dpa has been examined.

2.2 Experimental Procedure

The chemical composition of F82H used in this study is shown in Table 2.1. The specimens were first normalized at 1040°C for 0.5 h and tempered at 750°C for 1 h. A second heat treatment was performed on the F82H steel, which was secondly normalized at 1040°C for 0.5 h and tempered at temperatures of 750, 780 and 800°C for 0.5 h. The tempering time at 750°C was varied between 0.5 and 10 h. SS-3 tensile specimens were prepared from the normalized and tempered F82H. The SS-3 sheet tensile specimens were 0.76 mm thick with a gage length of 7.62 mm and 1.55 mm in width. Irradiation was carried out in the Japan Materials Test Reactor (JMTR) to neutron fluences of 1.2×10^{21} n/cm² ($E > 1$ MeV) and 2.2×10^{21} n/cm² ($E < 0.5$ MeV), resulting in a displacement damage value of 1.9 dpa. Capsule 00M-61A, nominally at 250°C, was a shrouded type with reactor coolant flowing over an aluminum cladding tube containing the specimens; gamma-heating was used to raise the specimen temperature. After irradiation, tensile testing was carried out in air at 250°C at a strain rate of 4.4×10^{-4} s⁻¹.

2.3 Microstructures of F82H Steels before Irradiation and Tensile Properties of F82H Steel irradiated at 250°C

Figs. 2.1(a)-2.1(c) show microstructures of F82H tempered at 750, 780 and 800°C for 0.5 h, respectively. Dislocation densities decreased with increasing tempering temperature. The size of $M_{23}C_6$ carbides increased with it and the number density in the matrix decreased with it as seen in Fig. 2.1(b) and 2.1(c). Figs. 2.2(a) - 2.2(c) show the microstructures of F82H tempered at 750°C for 1, 2 and 10 h, respectively. Dislocation densities decreased with increasing tempering time. The size of $M_{23}C_6$ carbide of F82H tempered at 750°C increased with the tempering time from 1 h to 10 h.

The changes of tensile properties with tempering temperature and time in F82H steel irradiated at 250°C to 1.9 dpa are shown in Figs. 2.3 and 2.4. The yield stress of F82H steel tempered at 780 and 800°C was lower than that tempered at 750°C before irradiation, but the former was higher than the latter after irradiation as given in Fig. 2.3. The yield stress of F82H steel tempered at 750°C before irradiation decreased with increasing the tempering time. After the irradiation, the yield stress did not increase linearly with it as seen in Fig. 2.4. The yield stress of F82H tempered at different conditions is shown in Fig. 2.5, and the yield stress of F82H tempered at 750°C was smaller than those tempered at higher temperatures. The ability to strain harden was low for the F82H (UE: from 0.3 to 0.4%). The fracture surfaces after the tensile testing are shown in F82H steels tempered at 750, 780 and 800°C for 0.5 h, respectively, as shown in Figs. 2.6(a) - 2.6(c), and the reduction area of F82H tempered at 750°C is somewhat larger than the others.

Microstructures of martensitic steels, such as dislocations, carbides and lath width, depend strongly on the temperature and time of tempering. The density of dislocations of F82H decreased with increasing temperature and increasing time of tempering [10]. The concentration of carbon in solution in the matrix will increase with tempering temperature and time. In the specimen tempered at higher temperatures, the sink density for point defects due to dislocations becomes lower and the formation of dislocation loops may increase and the growth rate of dislocation loops will increase. The mobility of interstitial atoms in martensitic steels will be reduced with increasing an concentration of carbon in solution, and the formation of dislocation loops will also increase. Radiation-hardening can be evaluated by Orowan's theory for athermal bowing of dislocations around obstacles on a slip plane [11-13] and depends on the number density and size of defect clusters. The changes of sink density and the concentration of carbon in solution can affect the formation and growth of dislocation loops, and therefore, radiation hardening should depend on the temperature and time of tempering.

2.4 Summary

Microstructures and tensile properties of a martensitic steel F82H (Fe-8Cr-2W-0.1C-0.2V-0.04Ta) were examined as a function of time and temperature of tempering. A heat treatment was performed at temperatures from 750 to 800°C for 0.5 h after the normalizing at 1040°C for 0.5 h. The tempering time at 750°C was varied from 0.5 to 10 h. The tensile specimens were irradiated at 250°C to a neutron dose of 1.9 dpa in the JMTR (Japan Materials Testing Reactor), and tensile tests were carried out at 250°C after the irradiation. The microstructures of the non-irradiated specimens were observed by a transmission electron microscope. The density of dislocations decreased with increasing time and temperature of the tempering, and the size of $M_{23}C_6$ carbide increased with it. While the yield stress of the non-irradiated specimens decreased with increasing time and temperature of tempering, the yield stress of the irradiated specimens tended to increase with increasing temperature of the tempering. The yield stress of the irradiated F82H steel changed from about 525 to 750 MPa and depended on the conditions of tempering treatment before irradiation.

2.5 References

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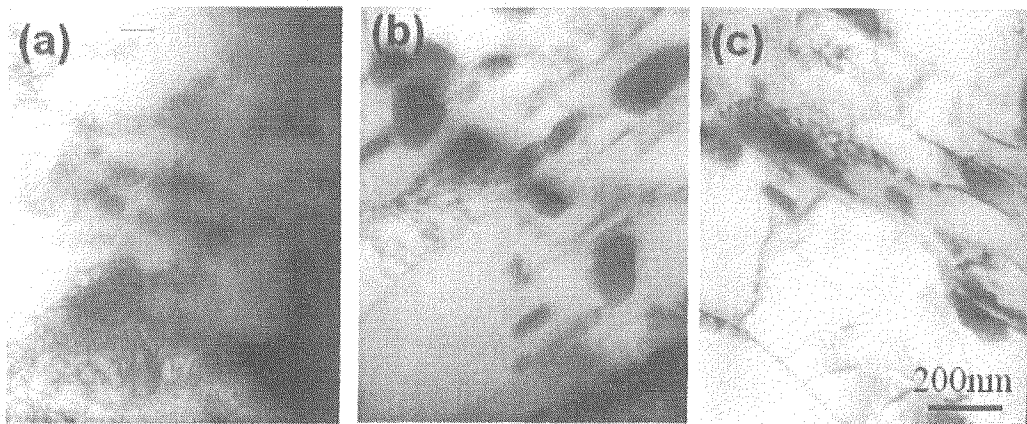


Fig. 2.1 Microstructures of F82H tempered at (a) 750°C, (b) 780°C, (c) 800°C, for 0.5 h. Dislocations and $M_{23}C_6$ carbides can be seen.

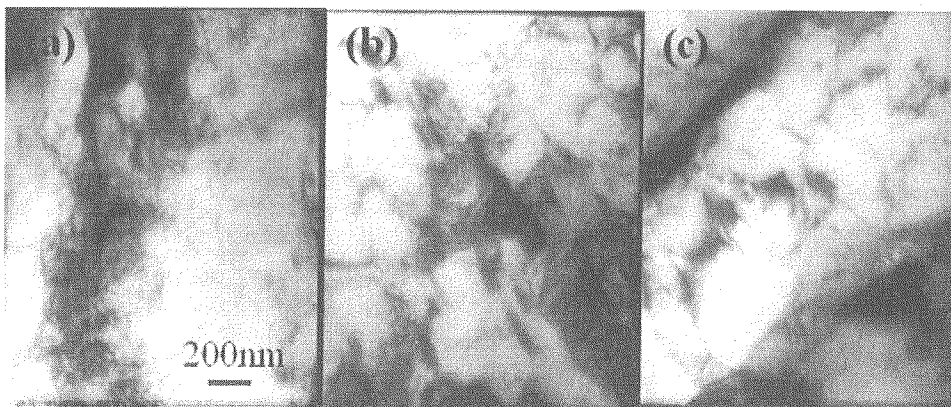


Fig. 2.2 Microstructures of F82H tempered at 750°C for (a) 1 h, (b) 2 h, (c) 10 h.

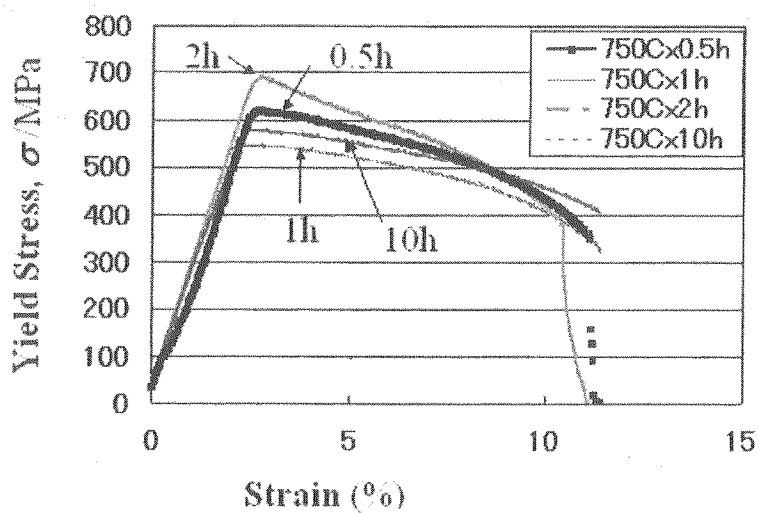


Fig. 2.3 Tensile curves of F82H tempered at 750, 780 and 800°C for 0.5 h and irradiated at about 250°C to 1.9 dpa in the JMTR. The tensile testing was performed at 250°C under a strain rate of $4.4 \times 10^{-4} \text{ s}^{-1}$.

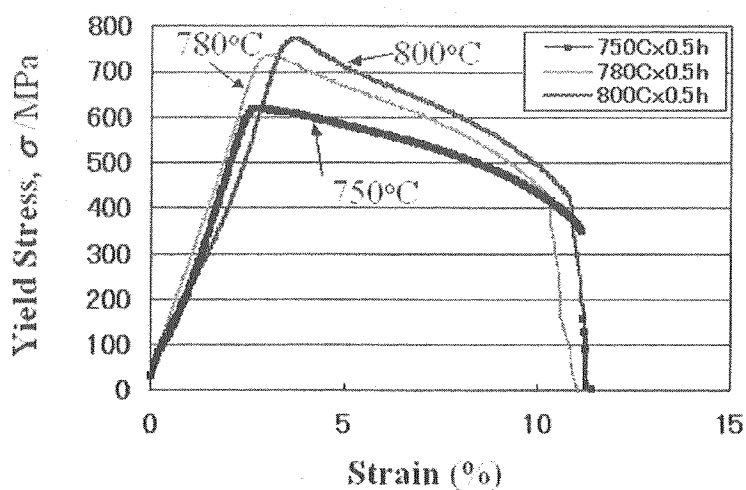


Fig. 2.4 Tensile curves of F82H, tempered at 750°C for 1 h, 2 h and 10 h, irradiated at about 250°C to 1.9 dpa in the JMTR.

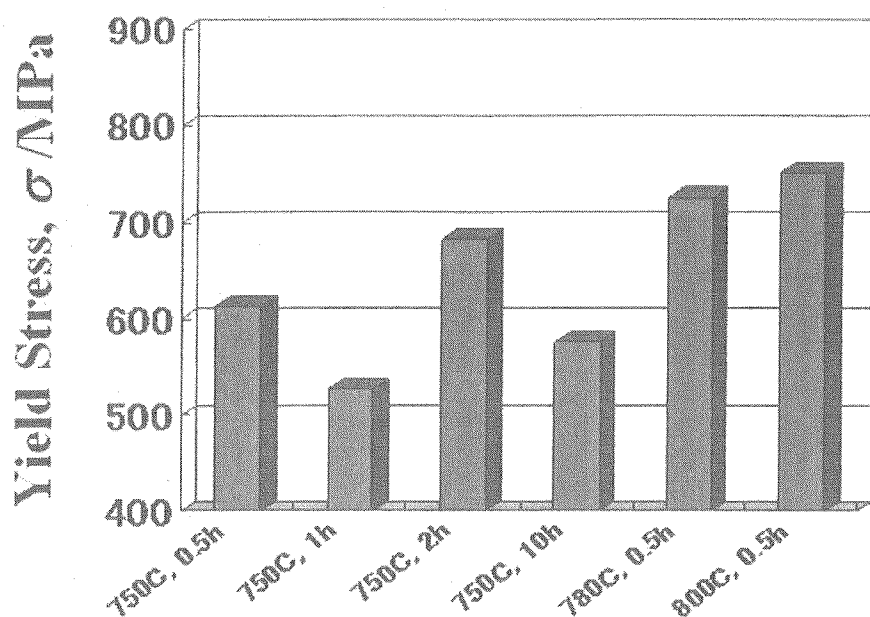


Fig. 2.5 Yield stress of the irradiated F82H tempered at different conditions.

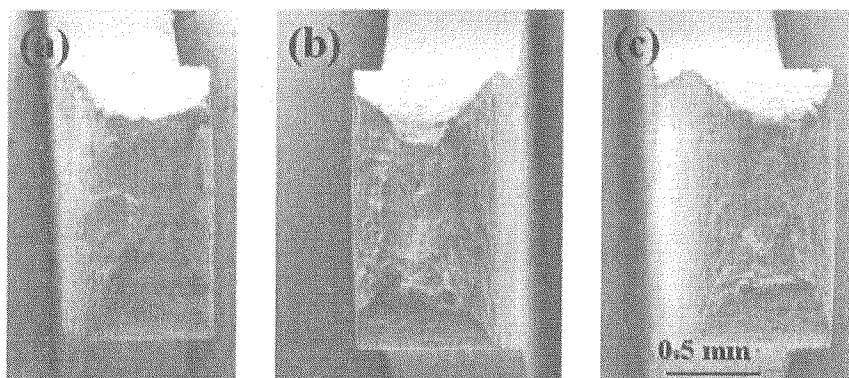


Fig. 2.6 The fracture surfaces after the tensile testing in F82H steels tempered at (a) 750°C for 0.5 h, (b) 780°C for 0.5 h and (c) 800°C for 0.5 h.

Table 2.1: Chemical composition of F82H used in this study (mass%).

Alloy	C	Sol. Al	Si	Mn	P	S	V	Ti	Cr	Ni
F82H	0.09	0.001	0.07	0.1	0.003	0.001	0.19	0.004	7.82	0.02

Alloy	Cu	Nb	Ta	W	B	O	N
F82H	0.01	0.0002	0.04	1.98	0.0002	-	0.007

3. Effect of Initial Heat Treatment on DBTT of F82H Steel Irradiated at 150 and 250°C by Neutrons

3.1 Research Background and The Purpose of This Study

Reduced-activation ferritic/martensitic steels are candidate materials for the blanket structure of fusion reactors. Radiation-hardening of 8-9%Cr martensitic steels irradiated by neutrons occurs mainly at irradiation temperatures lower than about 400°C, and it increases with decreasing irradiation temperature down to 250°C [1,2]. The shift of DBTT (ductile-brittle transition temperature) also increases with decreasing irradiation temperature, and the shift increases largely for irradiation at 250°C [3,4]. Several researchers [1-7] reported that the increase of yield strength and the shift of DBTT were different in several martensitic steels, such as F82H, JLF-1, JLF-1B, ORNL 9Cr-2WVTa, OPTIFER Ia, II, MANET II and Mod.9Cr-1Mo, which had different concentrations in some elements and were tempered at different temperatures. The effects of the normalizing and tempering of heat treatment on tensile and impact behavior in martensitic steels before irradiation were reported by L. Schafer [8] and P. Gondi [9]. However, the mechanisms of the changes of yield strength and DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment and impurities on them. The optimum heat treatment will be required to improve resistance to radiation hardening [10,11] and embrittlement. In this study, the dependence of impact properties on tempering time and temperature has been examined for martensitic steel F82H (Fe-8Cr-2W-0.2V-0.04Ta-0.1C) irradiated by neutrons.

3.2. Experimental Procedure

The chemical composition of F82H used in this study is shown in Table 3.1. The specimens were first austenitized at 1040°C for 0.5 h followed by air cool and tempered at 750°C for 1 h. A second heat treatment was performed on the F82H steel, which was austenitized at 1040°C for 0.5 h and tempered at temperatures of 750, 780 and 800°C for 0.5 h. The tempering time at 750°C was varied between 0.5 and 10 h. Miniaturized Charpy V-notched (CVN) impact (3.3 mm x 3.3 mm x 23.6 mm) specimens were fabricated. The 1/3CVN specimens were prepared from the normalized-and-tempered F82H steel. Irradiation was carried out in the Japan Materials Test Reactor (JMTR) of JAERI to neutron fluences of 1.2×10^{21} n/cm² ($E > 1$ MeV) and 2.2×10^{21} n/cm² ($E < 0.5$ MeV), resulting in a displacement damage value of 1.9 dpa. Capsules 00M-61A, nominally at 250°C, and 00M-62A, nominally at 150°C, were a shrouded type with reactor coolant flowing over an aluminum cladding tube containing the specimens; gamma-heating was used to raise the specimen temperature. After irradiation, Charpy impact testing was carried out in the hot cell of the JMTR of JAERI, and the absorbed energy was measured as a function of temperature.

3.3 Impact Properties and Tensile Properties of F82H Steels Irradiated at 150 and 250°C

Impact energies as a function of temperature for the 1/3CVN F82H specimens before irradiation and after irradiation at 250°C to 1.9 dpa are shown in Figs. 3.1 and 3.2, respectively. DBTT of the specimens ranged from -118 to -94°C before irradiation [12]. The DBTTs of the specimen tempered at 780°C for 0.5 h and 750°C for 10 h increased slightly as seen in Fig. 3.1. The DBTT after the irradiation ranged from -23 to 25°C. The effect of tempering time at 750°C for DBTT was not clear, but DBTT of the irradiated specimen tempered at 780°C was lower than that at 750°C, and the effect of tempering temperature on DBTT was relatively large. In Fig. 3.3, the impact energies as a function of temperature for the 1/3CVN F82H specimens irradiated at 150°C to 1.9 dpa are also shown. In this case, these DBTTs ranged from 0 to 15°C, and they were very similar to each other. Mechanical properties of F82H steel becomes very sensitive to irradiation temperatures at ~250°C (and higher). The difference of DBTT behavior between 150 and 250°C irradiation specimens may be due to the difference of defect clusters formed by irradiation, depending on migrations of point defects, or the difference of deformation mode such as dislocation channeling.

Figure 3.4 shows tensile curves of F82H specimens irradiated at 250°C to 1.9 dpa [10]. The tensile specimens were tempered at different conditions before irradiation. The strengths and elongations of the specimens before and after irradiation depended on the tempering conditions. Figure 3.5 shows yield stress of the specimens before and after irradiation. The radiation hardening depended on the tempering conditions, and it increased with increasing time and temperature of tempering before irradiation.

3.4 Relation of DBTT and Yield Stress or Δ DBTT and Δ YS

The relation of DBTT and yield stress before and after irradiation in the F82H specimens tempered at different conditions is shown in Fig. 3.6. DBTT of the specimens before irradiation seems to decrease slightly with increasing yield stress as seen Fig. 3.6(a). In the higher DBTT indicated by dotted circles, larger size carbides were formed in the specimens [11], and DBTT might be affected by the formation of larger size carbides. DBTT after irradiation tended to decrease with increasing yield stress as shown in Fig. 3.6(b). Figure 3.7 shows the relation of Δ DBTT and Δ YS due to irradiation in F82H steels irradiated at 250°C to 1.9 dpa. The ratios of Δ DBTT to Δ YS are from 0.2 to 1.0 °C/MPa, and the values tended to decrease with increasing time and temperature of tempering. In application of heat treatment of tempering for ferritic/martensitic steels, lower DBTT during irradiation is desired for a long life of fusion reactors, and 780°C tempering can be better condition because of a smaller ratio of Δ DBTT to Δ YS.

The initiation of a cleavage event in the fracture toughness is generally determined by the magnitude of the normal tensile stress, which has to exceed a critical value over a finite region ahead of the crack tip. When radiation hardening is of sufficient magnitude for this to occur, cleavage will be initiated. It is possible that inhomogeneous channel deformation could lead to a reduction in the critical fracture stress through the initiation of micro-cracks at dislocation pile-ups. There is a possibility that the irradiated

specimens tempered at lower temperature and shorter time before irradiation may be easily inhomogeneously deformed under dislocation channels and the critical fracture stress could be reduced. As reported in ref. [13], the pre-irradiation microstructure could affect the post-irradiation deformation mode. In this study, if the pre-irradiation microstructures are also different under the tempering conditions, then the deformation mode would be affected. The difference of the deformation behavior may be also related to amounts of segregation of solute atoms to defect sinks. As a result, the relation of ΔDBTT to ΔYS as given in Fig. 3.7 might be obtained.

3.5 Summary

The dependence of ductile-brittle transition temperature (DBTT) on tempering time and temperature was examined for a martensitic steel F82H irradiated at 150 and 250°C to a neutron dose of 1.9 dpa in the JMTR. The heat treatment was performed at 750 and 780°C for 0.5 h after the normalizing at 1040°C for 0.5 h. The tempering time at 750°C was varied from 0.5 to 10 h. 1/3CVN specimens were used in this study, and the absorbed energies in the impact tests were measured as a function of temperature. DBTT of F82H steels irradiated at 250°C to 1.9 dpa was ranged from -23 to 25°C, and DBTT of F82H steels irradiated at 150°C to 1.9 dpa was ranged from 0 to 15°C. DBTT of F82H steels irradiated at 250°C depended strongly on temperature and time of tempering, and it tended to decrease with increasing yield stress. The effect of tempering conditions on DBTT was smaller in the specimens irradiated at 150°C. ΔDBTT due to irradiation in the F82H steels irradiated at 250°C tended to decrease with increasing time and temperature of tempering.

3.6 References

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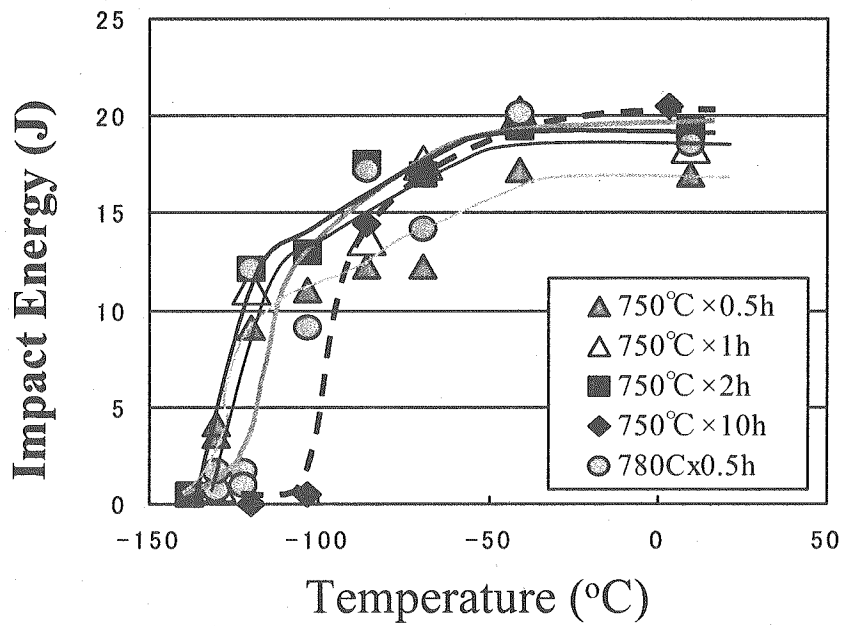


Fig. 3.1 Impact energies as a function of temperature of F82H steels before irradiation [12]. The specimens were tempered at 750°C for 0.5, 1, 2, and 10 h and 780°C for 0.5 h.

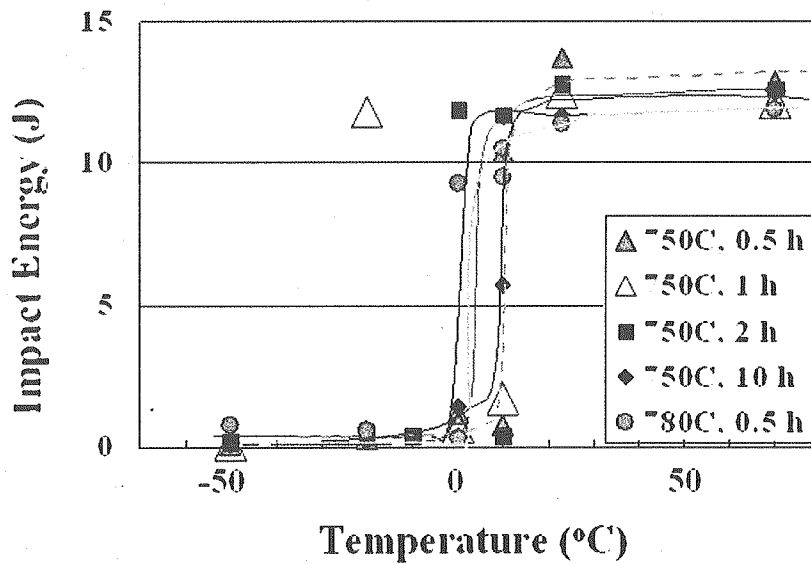


Fig. 3.2 Impact energies as a function of temperature of F82H steels irradiated at 250°C to 1.9 dpa. The specimens were tempered at 750°C for 0.5, 1, 2, and 10 h and 780°C for 0.5 h before irradiation.

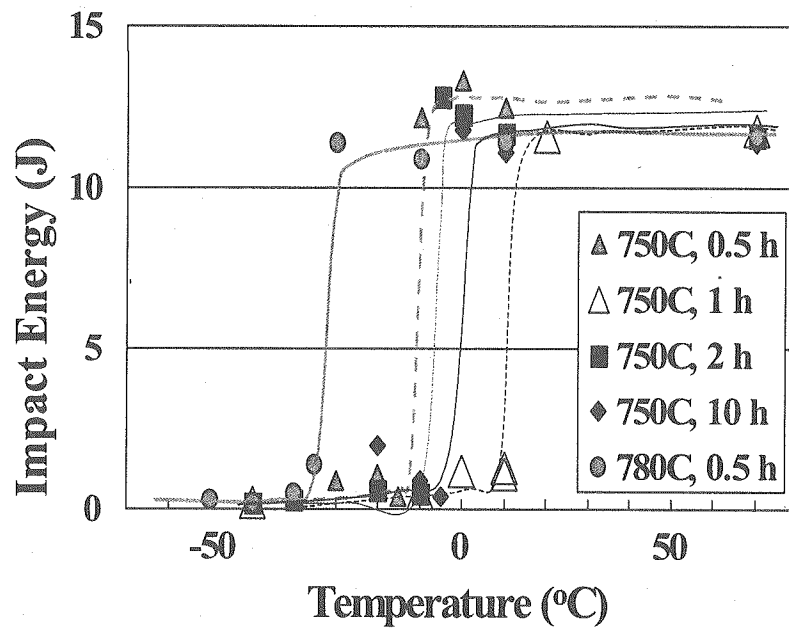


Fig. 3.3 Impact energies as a function of temperature of F82H steels irradiated at 250°C to 1.9 dpa. The specimens were tempered at 750°C for 0.5, 1, 2, and 10 h and 780°C for 0.5 h before irradiation.

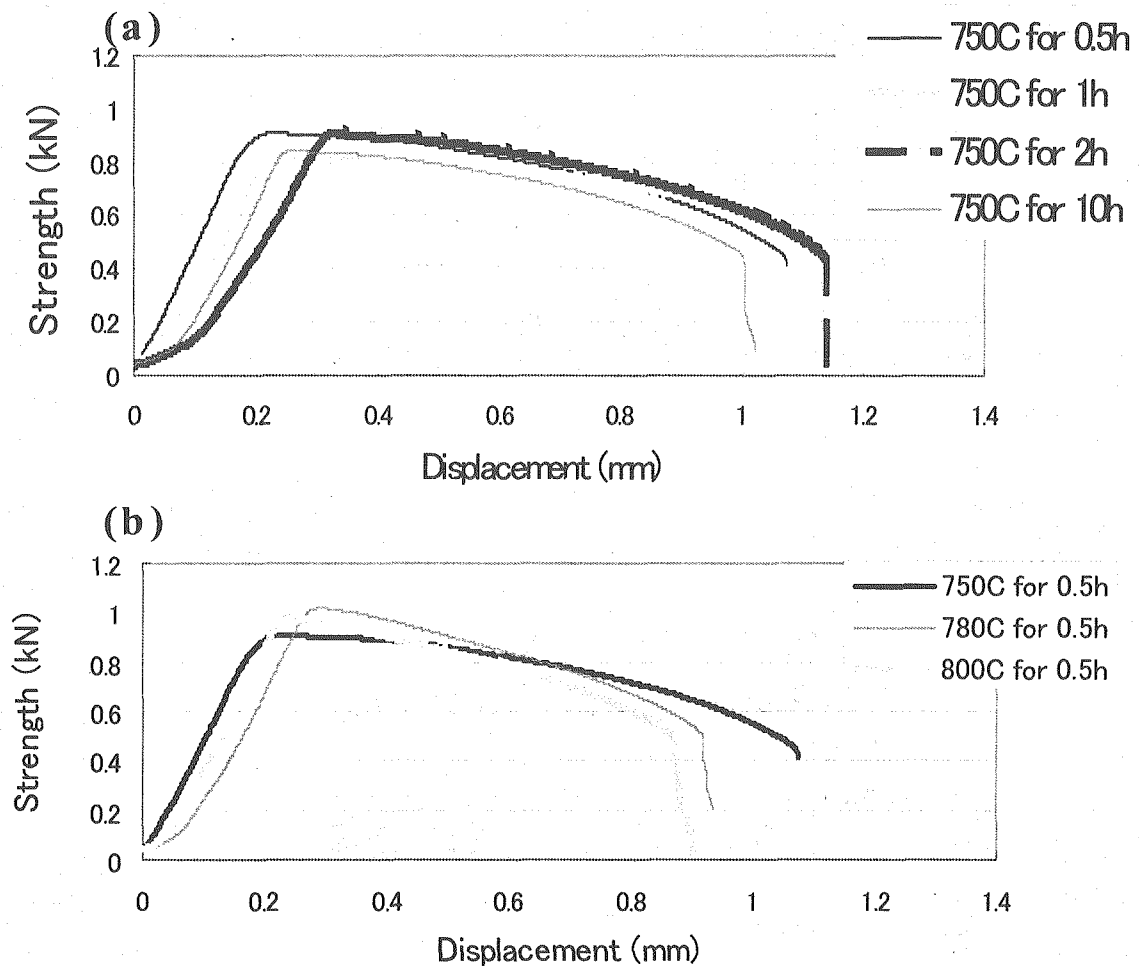


Fig. 3.4 Tensile curves of F82H steels irradiated at 250°C to 1.9 dpa. The specimens were tempered at 750°C for 0.5, 1, 2 and 10 h (a) and 750, 780 and 800°C for 0.5 h (b). These tensile data were taken from ref. [10].

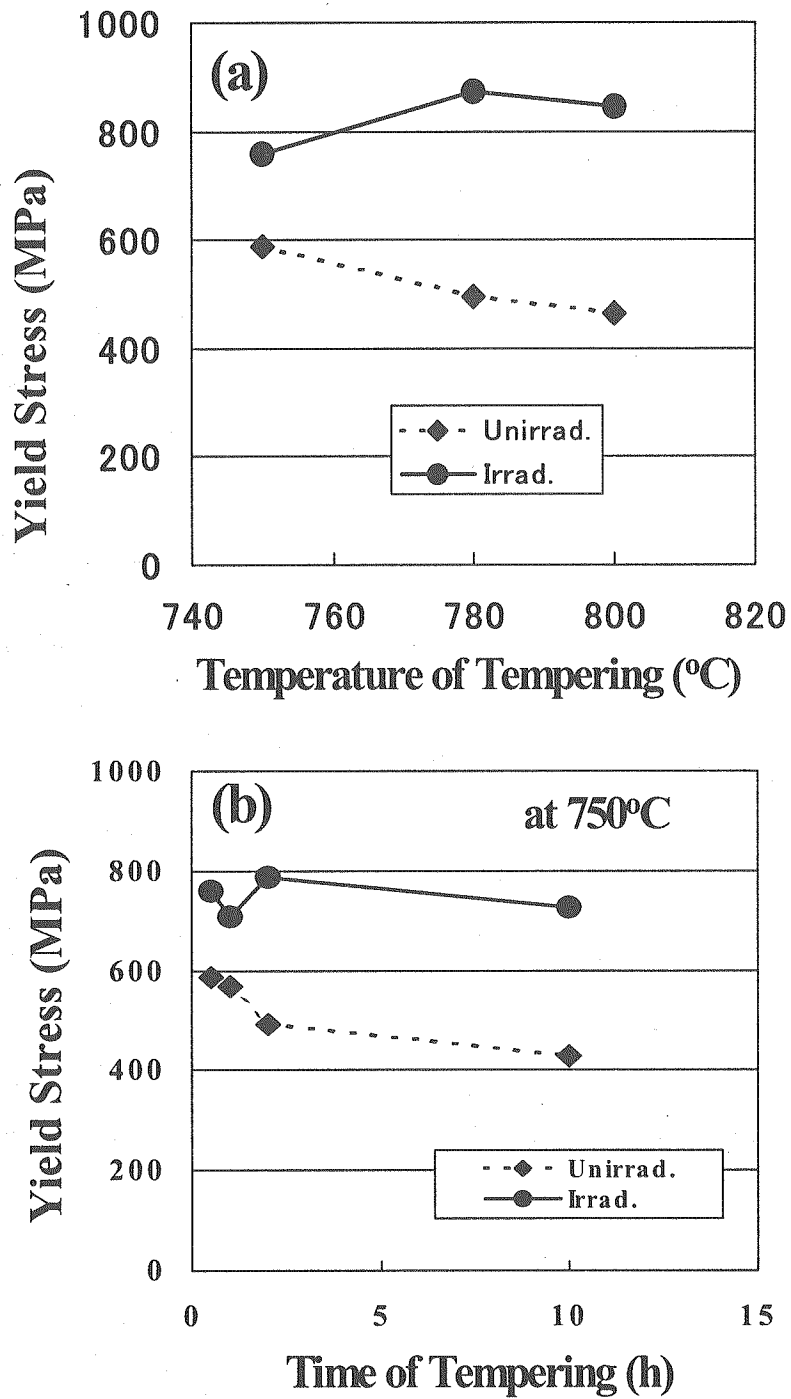


Fig. 3.5 Changes of yield stress of F82H steel irradiated at 250°C to 1.9 dpa. Tensile data were taken from ref. [10].

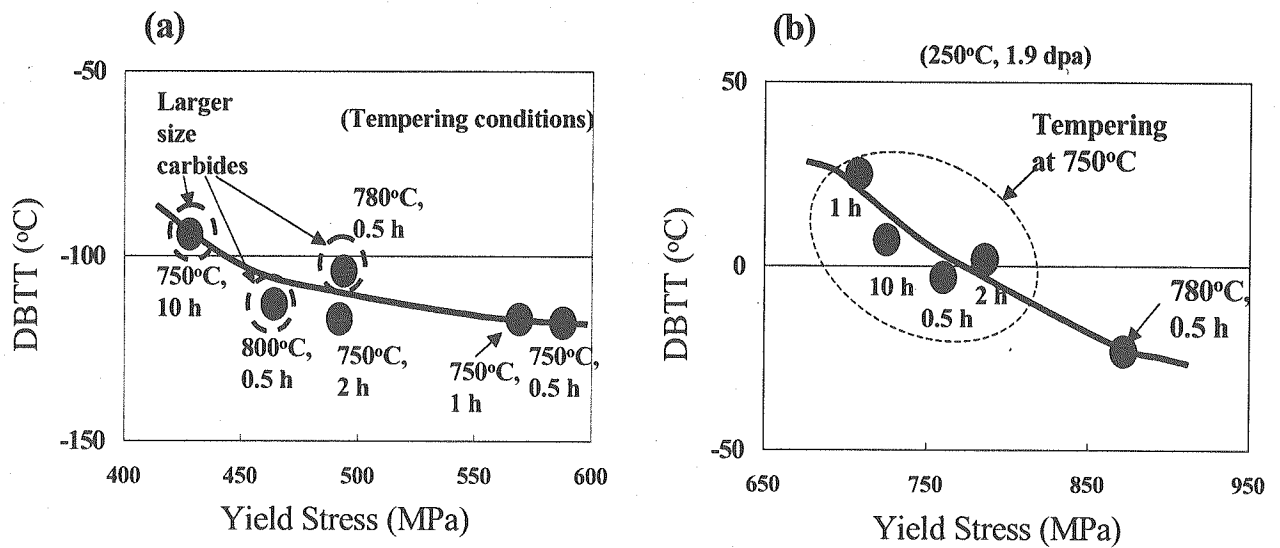


Fig. 3.6 Relation of DBTT and yield stress of F82H tempered with different conditions before (a) and after (b) irradiation. Data of DBTT for the unirradiated specimens were taken from ref. [12].

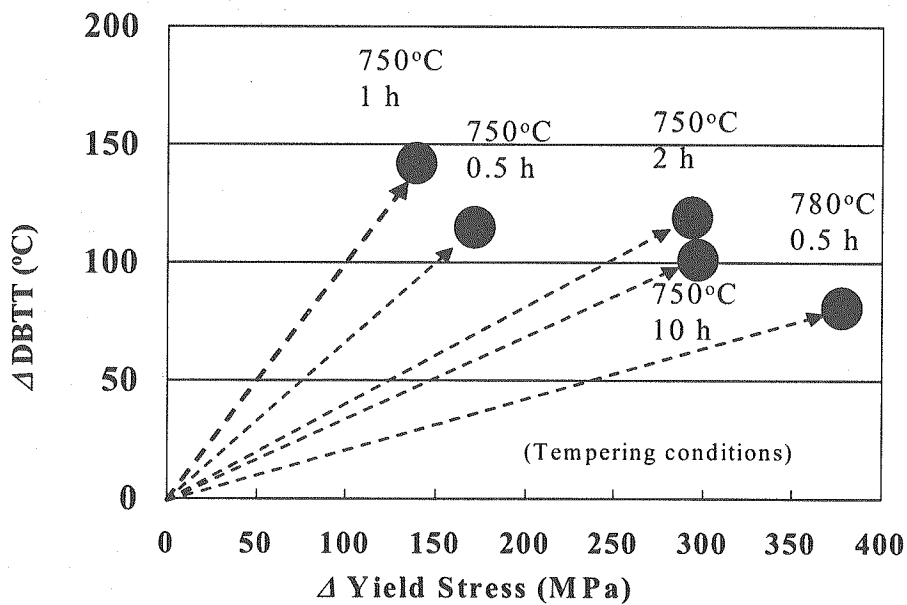


Fig. 3.7 Relation of Δ DBTT and Δ YS due to irradiation at 250°C to 1.9 dpa in F82H steels tempered with different conditions.

Table 3.1: Chemical composition of F82H steel used in this study (wt%).

Alloy	C	Sol. Al	Si	Mn	P	S	V	Ti	Cr	Ni
F82H	0.09	0.001	0.07	0.1	0.003	0.001	0.19	0.004	7.82	0.02

Alloy	Cu	Nb	Ta	W	B	O	N
F82H	0.01	0.0002	0.04	1.98	0.0002	-	0.007

4. Effects of Helium Production and Heat Treatment on Neutron Irradiation Hardening of F82H Steels Irradiated with Neutrons

4.1 Research Background and The Purpose of This Study

Reduced-activation ferritic/martensitic steels are candidate materials for the vessel of the liquid target of spallation neutron sources and for the blanket structure of fusion reactors. Structural materials for these applications must withstand not only displacement damage but also transmutation-induced embrittlement. Therefore, the effects of neutron irradiation on tensile deformation, ductile to brittle transition temperature (DBTT), and microstructures of F82H and the other ferritic/martensitic steels have been extensively investigated.¹⁻¹³⁾ On the course of these research activities, hardening and upward shift of DBTT induced by neutron bombardment have been commonly regarded as crucial problems. Radiation hardening occurs mainly at irradiation temperatures lower than about 400°C, and it increased with decreasing irradiation temperature down to about 250°C. The DBTT tends to increase with decreasing irradiation temperature as well, and the shift increases largely at 250°C. The issue of helium accumulation effects on these properties has been an ongoing concern. Recently, the effect of helium production on radiation hardening has been examined and large enhancement of hardening due to helium from 600 appm to 5000 appm was detected in tensile tests on 9Cr martensitic steels EM10 and T91 implanted by cyclotron experiments.^{14,15)} Slight enhancement of radiation hardening was also reported in neutron irradiation experiments^{16,17)} and other ion beam experiments.^{18,19)} In the other cyclotron experiments,^{20,21)} the enhancement of radiation hardening due to helium production was hardly observed up to 600 appm He. The studies on DBTT shift due to helium have been done by several researchers.⁸⁻¹⁰⁾ However, the details of the dependence of DBTT on helium production has not been made clear yet. Radiation-induced degradation of fracture toughness related to helium production is recognized to be one of the critical issues of the alloys. Furthermore, the synergistic effects of helium, hydrogen and displacement damage have been reported for swelling behavior²²⁻²⁴⁾ and mechanical properties²⁵⁻³¹⁾, and the synergistic effect has been another ongoing concern.

In order to clarify the influence of helium production on radiation hardening, the isotope ¹⁰B doping technique was used to introduce helium atoms in the alloy. The amount of helium was varied with using different ¹⁰B concentrations in a mixed spectrum reactor irradiation with thermal and fast neutrons. Since doping of the B element can affect the mechanical properties before and after irradiation, the isotope effect of B was minimized by comparing the results of mechanical tests of ¹⁰B and ¹¹B doped specimens. One of purpose in this study is to evaluate quantitatively the contribution of helium production to radiation hardening in F82H as a function of irradiation temperature and helium concentration, considering the synergistic effect of helium and displacement damage by means of ¹⁰B -method.

Several researchers^{6,8-10,32-34)} reported that the increase of yield strength and the shift of DBTT were different in several martensitic steels, such as F82H, JLF-1, JLF-1B, ORNL 9Cr-2WVTa, OPTIFER Ia, II, MANET II and Mod.9Cr-1Mo, which had different concentrations in some elements and were tempered at different temperatures. The effects of the normalizing and tempering of heat treatment on tensile and

impact behavior in martensitic steels before irradiation were reported by L. Schafer and P. Gondi.^{35,36)} However, the mechanisms for the relation between the changes of yield strength and shift of DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment and impurities on them. The optimum heat treatment will be required to improve resistances to radiation hardening and embrittlement. Another of purpose in this study is to present the effect of tempering conditions on irradiation hardening and shift of DBTT in F82H-std steel.

4.2 Experimental Procedure

The materials used in this study are F82H-std and F82H doped with ^{10}B , $^{10}\text{B}+^{11}\text{B}$ and ^{11}B . Their compositions are given in Table 4.1. In order to produce helium atoms the materials were doped with about 60 mass ppm B. The purity of isotope elements of ^{10}B and ^{11}B used in this study was about 95%. The 15 mm thick plates of F82H+B steels were normalized at 1040°C for 40 minutes in air followed by air cooling and tempered at 750°C for 60 minutes in air. Heat treatments for the 25 mm thick plates of F82H-std (IEA-heat, ID: 31W-11) were firstly performed at 750°C after the normalizing at 1040°C for 0.63 h in air followed by air cooling (single treatment). Second normalizing for the 5 mm thick blocks of F82H-std was carried out in a vacuum at 1040°C for 0.5 h followed by air cooling, and the time of subsequent tempering at 750°C was varied from 0.5 to 10 h in a vacuum. The second tempering was also performed at 780°C for 0.5 h in a vacuum (dual treatment). SS-3 specimens were used to evaluate the hardening in this study. The SS-3 tensile specimens were 0.76 mm thick with a gage length of 7.6 mm as shown in Fig. 4.1 and were taken parallel to the rolling direction.

Irradiation for the F82H+B specimens was carried out nominally at 300°C in the capsule 00M-65A of the Japan Materials Test Reactor (JMTR) in the Japan Atomic Energy Research Institute (JAERI) to neutron fluences of $1.4 \times 10^{21} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) and $1.2 \times 10^{21} \text{ n/cm}^2$ ($E < 0.683 \text{ eV}$), resulting in a displacement damage of $\sim 2.2 \text{ dpa}$. The averaged displacement damage induced by the reaction of $^{10}\text{B}(n, \alpha)^7\text{Li}$ was calculated as about 0.2 and 0.1 dpa for the F82H+ ^{10}B and F82H+ $^{11}\text{B}+^{10}\text{B}$, respectively. The total displacement damage due to the transmutation reaction and neutron irradiation was 2.4 dpa in the F82H+ ^{10}B .

Irradiation for the F82H-std and F82H+B specimens was also performed nominally at 150°C in the capsule 00M-62A of JMTR to $\sim 1.9 \text{ dpa}$. The concentrations of helium in the specimens, which was generated through $^{10}\text{B}(n, \alpha)^7\text{Li}$ reaction, after the irradiations were measured using a mass analyzer of magnetic reflection type. In this measurement, helium gas was released into the measurement system from the ion-implanted specimen after melting. In this technique, a known concentration of He gas is used for a standard relation between the gas intensity and concentration of He. The helium concentrations of F82H+ ^{11}B , F82H+ $^{10}\text{B}+^{11}\text{B}$ and F82H+ ^{10}B steels were about 15, 190 and 330 appm, respectively.

After neutron irradiation, tensile tests were performed. Tensile testing was carried out in a vacuum at a strain rate of $4 \times 10^{-4} \text{ s}$ at 20, 300, and 400°C in the JMTR hot laboratory.

4.3 Tensile Properties of F82H Steels

4.3.1 Effect of Helium Production on Tensile Properties Irradiated F82H steels

Tensile tests of F82H+¹⁰B, F82H+¹¹B, and F82H+¹⁰B+¹¹B specimens were performed at 300°C after irradiation. The increments of yield stress, ΔYS , and ultimate tensile strength, ΔUTS , due to irradiation are given in Fig. 4.2. In the F82H+¹⁰B+¹¹B and F82H+¹⁰B, the ΔYS and ΔUTS were very smaller than the F82H+¹⁰B. The summary of tensile data is given in Table 4.2.

Tensile tests of F82H+¹⁰B, F82H+¹¹B, and F82H+¹⁰B+¹¹B specimens irradiated at 150°C to 1.9 dpa were performed at 20°C. The YS and ΔUTS are given in Fig. 4.3. No significant increment of radiation hardening due to helium production was observed in this condition. The summary of tensile data is shown in Table 4.3.

In Figs. 4.2 and 4.3, ΔYS was larger in ¹⁰B doped steel irradiated at 300°C to 2.3 dpa, but no increment was observed in the steel irradiated at 150°C to 1.9 dpa. No enhancement of radiation hardening at 150°C was similar to previous result of cyclotron experiment,²⁰⁾ and the slight enhancement of the hardening at 300°C was also similar to the previous works of ion irradiation experiments.^{18,19)} Thus, radiation hardening in the ¹⁰B(¹¹B) doped steels may depend on irradiation temperature. Radiation hardening is induced by the formation of defect clusters, and it can be evaluated by Orowan's theory for athermal bowing of dislocations around obstacles on a slip plane.^{38,39)} The formation and growth of defect clusters depends on irradiation parameters such as temperature, dpa, dpa rate and helium production rate,^{18,22,23,40,41)} and the microstructures of the irradiated F82H+¹⁰B, F82H+¹¹B, and F82H+¹⁰B+¹¹B specimens would be changed by irradiation, depending on irradiation temperature and helium production. More precious experiments are required to discuss the helium effect on radiation hardening.

4.3.2 Effect of Heat Treatment on Tensile Properties of F82H Steels Irradiated at 150°C

Fig. 4.4 shows tensile curves of F82H specimens that were heat-treated at single and dual normalizing and tempering (N&T) conditions and then irradiated at 150°C to 1.9 dpa. The tensile tests were performed at 20°C. The tensile data of F82H steels before irradiation for the single and dual N&T treatments are given in Table 4.4,⁴²⁾ and the tensile property of dual N&T treatments is very similar to that of single N&T treatments. However, radiation hardening of dual heated F82H steel was smaller and the total elongation was longer than that of the single heated N&T specimen. The cause of the hardening changes induced by the re-heat treatment may be related with the levels of materials homogeneity in F82H steel. The effects of tempering time and temperature in the re-heat treatment on tensile properties of F82H steels irradiated at 150°C to 1.9 dpa were also examined at 20°C. The summary of tensile data is shown in Table 4.3. Figs. 4.5(a) and 4.5(b) present the yield stress before and after irradiation, and the increment of yield stress due to irradiation, respectively, as a function of tempering time. The increment of yield stress

due to irradiation tended to increase with increasing tempering time.

The relation between the shift of DBTT and the increment of yield stress due to 150°C irradiation in F82H steels heated with different tempering time and temperature is given in Fig. 4.6(a). The ratios of Δ DBTT to Δ YS were in the range from 0.3 to 1.3 °C/MPa, and the values tended to decrease with increasing time and temperature of tempering. In previous studies, DBTT of the specimens before and after irradiation was formerly examined.^{43,44)} A similar result of the relation between Δ DBTT to Δ YS was reported for F82H-std irradiated at 250°C to 1.9 dpa as a function of time and temperature of tempering.⁴²⁾ Δ DBTT of F82H-std irradiated at 250°C tended to decrease with increasing time and temperature of tempering, and the ratios of Δ DBTT to Δ YS were in the range from 0.2 to 1.0°C/MPa as given in Fig. 4.6(b). In application of tempering for ferritic/martensitic steels, lower DBTT during irradiation is desired for a long life of fusion reactors, and tempering treatment with longer time or higher temperature seems to be better condition because of a smaller ratio of Δ DBTT to Δ YS. The tempering conditions are also important for the initial strength of structural materials of fusion reactor and we have to select the condition carefully.

The initiation of a cleavage event in the fracture toughness is determined by the magnitude of the normal tensile stress, which has to exceed a critical value over a finite region ahead of the crack tip. When radiation hardening is of sufficient magnitude for this to occur, a cleavage fracture will be initiated. It is possible that the localized channel deformation leads to a shearing rupture through the initiation of micro-cracks at dislocation pile-ups. As reported in a reference,⁴⁵⁾ the pre-irradiation microstructures could affect the post-irradiation deformation mode, since the irradiated specimens tempered at lower temperature and shorter time before irradiation may be easily inhomogeneously deformed under dislocation channels and the critical fracture stress could be reduced, resulting in the loss of strain-hardening capacity. In contrast, the deformation mode of the specimen tempered at higher temperature and longer time was not dislocation channels but deformation bands. The pre-irradiation microstructures, especially the number density of dislocation loops, were different in the specimens tempered at different conditions. The deformation mode could be affected by the pre-irradiation microstructures. On the other hand, it is reported that irradiation changes both the amount and chemical composition of precipitates in F82H, JLF-1 and ORNL-9Cr steels after the irradiation at 300°C to 5 dpa.⁴⁶⁾ In this study, the pre-irradiation microstructures are considered to be different after different tempering conditions, and the number density and size of precipitates will be furthermore changed by irradiation. Chemical concentrations at defect sinks would be changed by radiation-induced segregation (RIS). The deformation mode would be hence affected by the microstructural changes, the chemical compositions of precipitates and RIS. These may be the cause of different ratios between Δ DBTT and Δ YS given in Fig. 4.6.

4.4 Summary

The influence of ^{10}B and ^{11}B additions on radiation hardening was examined in a reduced-activation martensitic F82H+B steel (8Cr-2W-0.2V-0.04Ta-0.1C+0.006B). The specimens used were ^{10}B doped, $^{10}\text{B}+^{11}\text{B}$ doped and ^{11}B doped F82H steels. Increment of radiation hardening, which contains about 330 appm, in the ^{10}B (^{11}B) doped steels depends on irradiation temperature, although the displacement damage is not exactly same among the irradiations. The effect of ^{10}B addition on increment of radiation hardening was not observed for 150°C irradiation.

The dependence of radiation hardening on tempering conditions, time and temperature, was also examined for a F82H-std steel irradiated at 150°C to 1.9 dpa. Irradiation hardening of the F82H-std steel due to this irradiation depended on temperature and time of tempering, and it tended to increase with increasing tempering time and temperature. Obtained relationship between the shift of DBTT and the increment of yield strength due to irradiation implies that an extension of tempering time or increase of tempering temperature would bring about a preferable effect on DBTT shift of F82H-std.

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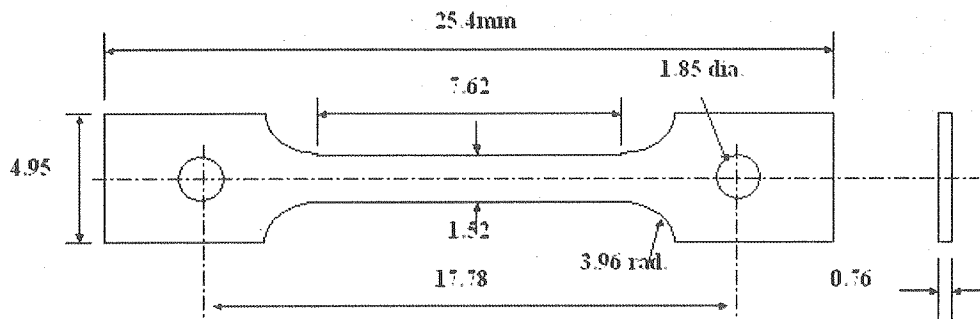


Fig. 4.1 Tensile specimen geometry of SS-3 type.

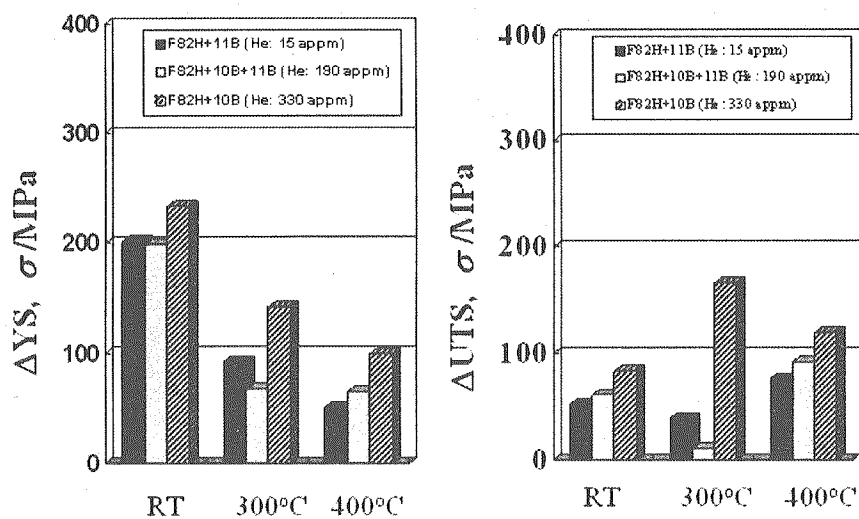


Fig. 4.2 Increments of yield stress and ultimate tensile stress of F82H+B steels irradiated at 300°C to 2.3 dpa. The tensile tests were performed at 20, 300 and 400°C.

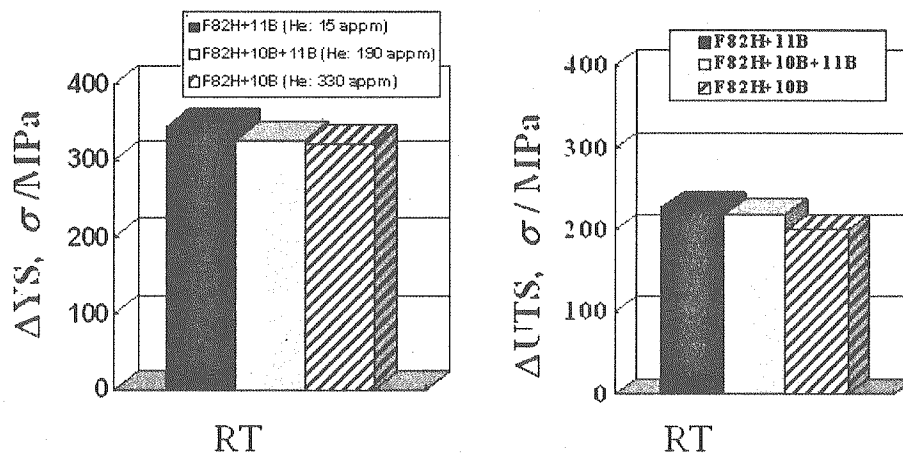


Fig. 4.3 Increments of yield stress and ultimate tensile stress of F82H steels irradiated at 150°C to 1.9 dpa. The tensile tests were performed at 20°C.

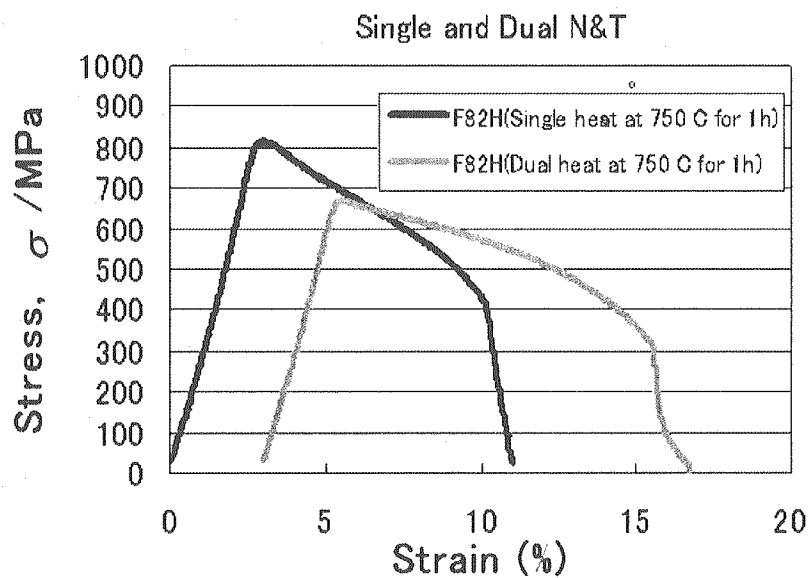


Fig. 4.4 Tensile curves of F82H specimens, heat-treated at single and dual N&T treatment conditions, and then irradiated at 150°C to 1.9 dpa. The tensile tests were performed at 20°C.

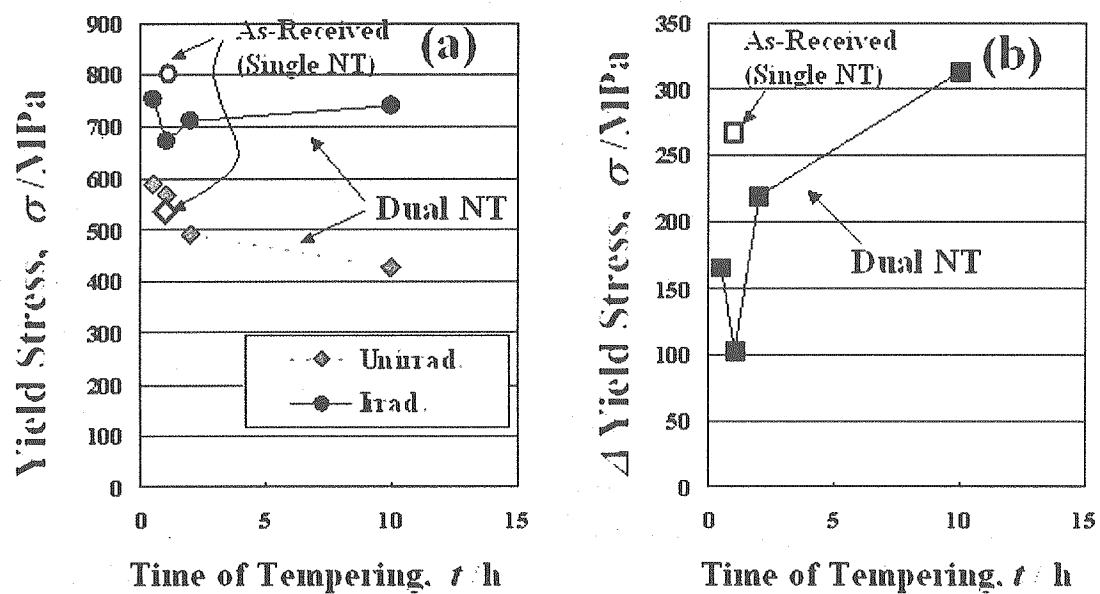


Fig. 4.5 Yield stress of the specimens before and after irradiation (a), and the increment of yield stress (b) induced by 150°C irradiation. The tensile tests were performed at 20°C.

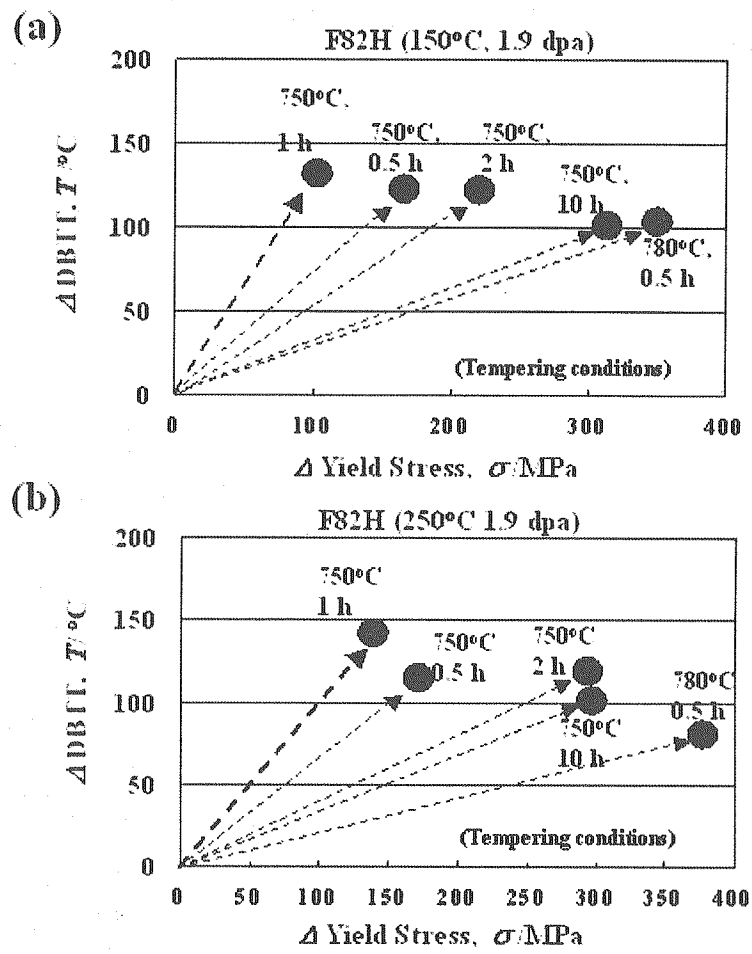


Fig. 4.6 Relationship between the shift of DBTT and the increment of yield stress induced by (a) 150°C and (b) 250°C ⁴³⁾ irradiation in F82H steels tempered with different time and temperature.

Table 4.1: Chemical compositions of F82H steels used in this study (wt%).

Materials	B	C	Si	Mn	P	S	Cr	W	V	Ta
F82H+ ¹⁰ B	0.0061	0.097	0.10	0.10	0.007	0.001	7.96	1.98	0.18	0.05
F82H+ ¹¹ B	0.0059	0.093	0.11	0.10	0.007	0.001	8.02	1.98	0.18	0.05
F82H+ ¹⁰ B+ ¹¹ B	0.0067	0.094	0.12	0.90	0.007	0.001	8.01	2.01	0.18	0.05
F82H	0.0002	0.09	0.07	0.1	0.003	0.001	7.82	1.98	0.19	0.04

Table 4.2: Tensile data of F82H+B steels irradiated at 300°C to 2.3 dpa in JMTR.

Alloys	He Production (appm)	Test Temperature (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	RA (%)	
F82H+ ¹¹ B	5	20	753	753	0.3	8.9	79	Ref. (37)
	5	300	563	563	0.4	9.7	81	Present data
	5	400	511	545	2.9	12.2	78	Present data
F82H+ ¹¹ B+ ¹⁰ B	190	20	788	796	0.3	8.8	73	Ref. (37)
	190	300	623	623	0.4	8.0	78	Present data
	190	400	511	545	2.9	12.2	78	Present data
F82H+ ¹⁰ B	330	20	796	805	0.4	8.9	70	Ref. (37)
	330	300	615	623	0.3	8.3	75	Present data
	330	400	563	589	2.1	10.2	62	Present data

Table 4.3: Tensile data of F82H and F82H+B steels irradiated at 150 °C to 1.9 dpa in JMTR. Some specimens were re-heated at different tempering conditions after the second normalizing at 1040°C for 0.5h. Tensile tests were performed at 20°C.

Alloys	He Production (appm)	Final Tempering Temperature (°C)	Final Tempering Time (h)	Re-heat N&T	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
F82H	2	750	0.5	Yes	754	758	0.3	10.8
F82H	2	750	1	Yes	672	672	0.2	11.4
F82H	2	750	1	No	800	813	0.2	8.8
F82H	2	750	2	Yes	712	712	0.2	11.8
F82H	2	750	10	Yes	741	741	0.2	11.3
F82H	2	780	0.5	Yes	839	839	0.3	10.2
F82H+ ¹¹ B	5	750	1	No	895	905	0.4	9.7
F82H+ ¹¹ B+ ¹⁰ B	190	750	1	No	915	923	0.3	9.0
F82H+ ¹⁰ B	330	750	1	No	885	886	0.3	9.1

Table 4.4: Tensile data of F82H steels for the single and dual N&T treatment before irradiation

Alloys	Final Tempering Temperature (°C)	Final Tempering Time (h)	Re-heat N&T	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	Ref.
F82H	750	1	No	548	652	5.5	15.9	(41)
F82H	750	1	Yes	569	663	5.5	16.6	(41)

5. Effect of Heat Treatments on Tensile Properties of F82H Steel Irradiated at 250°C by Neutrons

5.1 Research Background and The Purpose of This Study

Reduced-activation ferritic/martensitic steels are candidate materials for the blanket structure of fusion reactors. Irradiation hardening of 8-9%Cr martensitic steels irradiated by neutrons occurs mainly at irradiation temperatures lower than about 400°C, and it tends to increase with decreasing irradiation temperature down to around 250°C. The shift of DBTT also increases with decreasing irradiation temperature, and the shift increases largely for irradiation at 250°C. Several researchers [1-7] reported that the increase of yield strength and the shift of DBTT were different in Fe-9Cr alloy and several martensitic steels such as F82H, JLF-1, JLF-1B, ORNL 9Cr-2WVTa, OPTIFER Ia, II, MANET II and Mod.9Cr-1Mo, which had different concentrations in some elements and were tempered at different temperatures. The effects of the normalizing and tempering of heat treatment on tensile and impact behavior in martensitic steels before irradiation were reported by L. Schafer [8] and P. Gondi [9]. The increase of yield strength and the shift of DBTT due to irradiation were reported that these were different in several martensitic steels [3-5], which had different concentrations in some elements and were also tempered at different temperatures. However, the mechanisms for the relation between the changes of yield strength and shift of DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment and impurities on irradiation hardening and embrittlement [10-12]. The purpose of this study is to examine the mechanism of irradiation hardening of F82H steel, depending on tempering conditions and to measure also the test temperature dependence of tensile behaviors after irradiation.

5.2 Experimental Procedure

The chemical composition of F82H steel used in this study is shown in Table 5.1. In the JMTR experiment, the specimens were first normalized at 1040°C for 0.5 h and tempered at 750°C for 1 h. A second heat treatment was performed on the F82H steel, which was secondly normalized at 1040°C for 0.5 h and tempered at temperatures of 750, 780 and 800°C for 0.5 h. The tempering time at 750°C was varied between 0.5 and 10 h. SS-3 tensile specimens were prepared from the normalized and tempered F82H steel. The SS-3 sheet tensile specimens were 0.76 mm thick with a gage length of 7.6 mm. Irradiation was carried out in the Japan Materials Test Reactor (JMTR) to a displacement damage value of 1.9 dpa, nominally at 250°C. After the JMTR irradiation, tensile testing was carried out at 25, 250, 400 and 500°C at a strain rate of 4.4×10^{-4} /s. In the High Flux Isotope Reactor (HFIR) irradiation, the different heat treated IEA-F82H was prepared. The specimens were firstly normalized at 1040°C for 38 min and tempered at 750°C for 1h. The second heat treatments were performed at 800°C, 860°C and 920°C for 0.5 h, and continuously heated at 700°C for 10h, which were denoted as Mod 1-A, Mod 1-B and Mod 1-C, respectively. The HFIR experiments were also performed in the Rabbit capsule of the HFIR to a displacement damage value of 5

dpa at 300°C. The tensile tests after the HFIR irradiation was performed at 25°C under strain rates of $1.1 \times 10^{-3}/s$ and $1.1 \times 10^{-2}/s$.

5.3 Tensile Properties of F82H Steels Irradiated at 250°C

Figs. 5.1(a) and 5.1(b) show stress-strain curves of F82H steels irradiated at 250°C to about 2 dpa in JMTR. The specimens were tempered at 750°C for 1 h or 780°C for 0.5 h before irradiation. Yield stress (YS) was decreased with increasing test temperature, but the change of YS between 250°C and 400°C was relatively small. The yield stress of the specimen tempered at 780°C after the irradiation has a larger value than that at 750°C in the tests. Figs. 5.2(a) and 5.2(b) show YS of these F82H steels tested at 25 and 500°C before and after the irradiation, and the irradiation hardening of the specimens tempered at 780°C were larger than that tempered at 750°C in the tests. Figs. 5.3 and 5.4 show yield stress and elongations, respectively, measured at 25, 250, 400 and 500°C in F82H steels tempered at several different conditions. The irradiation hardening measured at 25°C in the specimens tempered at 750°C for 0.5 – 10 h was about 100 – 240 MPa, and that tempered at 780°C and 800°C for 0.5 h was about 300 MPa. In the tests at 500°C, the irradiation hardening of the specimens tempered at 750°C was almost lost, but that of the specimens tempered at 780 and 800°C was about 130 – 200 MPa. The dependence of elongations on tempering conditions was increased with increasing test temperature. Uniform elongations of these specimens measured at 25°C and 500°C were ranged within about 0.3~0.4% and 2~5%, respectively. The total elongations of these specimens measured at 25°C and 500°C were ranged within about 10-13% and 12~20%, respectively. Fig. 5.5 shows yield stress and ΔYS in four type heated F82H steels tested at 25°C, before and after the HFIR irradiation at 300°C to about 5 dpa. The Mod 1-A, -B and -C F82H steels had a larger ΔYS than that of IEA standard heat-treated F82H steel, and the elongations of Mod-1 groups after irradiation were slightly larger than that of IEA heat-treated F82H steel as given in Fig. 5.6. From these results, it is found that irradiation hardening of RAF's is very sensitive to tempering conditions, and the method of tempering heat conditions is very useful for the improvement of tensile properties after irradiation. Summaries of tensile data of unirradiated and irradiated specimens are given in Table 5.2 and 5.3, respectively.

Irradiation hardening is induced by the formation and growth of defect clusters, and it can be evaluated by Orowan's theory for athermal bowing of dislocations around obstacles on a slip plane [13,14]. In the study of tensile deformation, it was proposed that the deformation modes could be affected by the pre-irradiation microstructures [15]. As according to references of 16 and 17, it was reported that the irradiation changed both the amount and chemical composition of precipitates in F82H, JLF-1 and ORNL-9Cr steels after the irradiation at 300°C to 5 dpa. The formation and growth of defect clusters could depend on the initial microstructures and irradiation parameters such as temperature, dpa, dpa rate, and gas atoms [18-22]. The solubility of solute atoms such as carbon in matrix and the number densities of size of carbides would be strongly depended on tempering conditions. In higher temperature conditions of

tempering, carbon concentration in matrix will increase, and the mobility of interstitial atoms may be decreased and the densities and growth of dislocation loops will be changed. From these processes for the formation and growth of defect clusters, irradiation hardening of RAF's can be affected by the tempering conditions. Therefore, the method of heat treatment of tempering is very useful for the improvement of irradiation hardening and embrittlement.

5.4 Summary

The optimum heat treatment of reduced-activation ferritic steels (RAF's) is required to improve resistances to irradiation hardening and embrittlement, and effects of tempering conditions on tensile properties have been examined in F82H steel irradiated by neutrons. Irradiations were performed at 250°C in the JMTR to 2 dpa and at 300°C in the HFIR to 5 dpa. After the neutron irradiations, tensile tests were performed at temperatures from 25°C to 500°C. In the JMTR experiments, irradiation hardening measured at 25°C in the specimens tempered at 750°C for 0.5 – 10 h was about 100 – 240 MPa, and that tempered at 780°C and 800°C for 0.5 h was about 300 MPa. In the tests at 500°C, the irradiation hardening of the specimens tempered at 750°C was almost lost, but that of the specimens tempered at 780 and 800°C was about 130 – 200 MPa. These results may indicate that defect clusters formed in the specimens tempered at higher temperatures are relatively stable even at 500°C. In the HFIR experiments, the tensile properties of the F82H steels tempered at lower temperature of 700°C for 10 h after the heat treatments of 800, 860 and 920°C for 0.5 h were also examined, Irradiation hardening about 400 MPa in the specimens measure at 25°C was larger than that of IEA F82H standard heat treatment, but the elongations of the specimens were slightly better than that of IEA F82H-std heat treatment.

5.5 References

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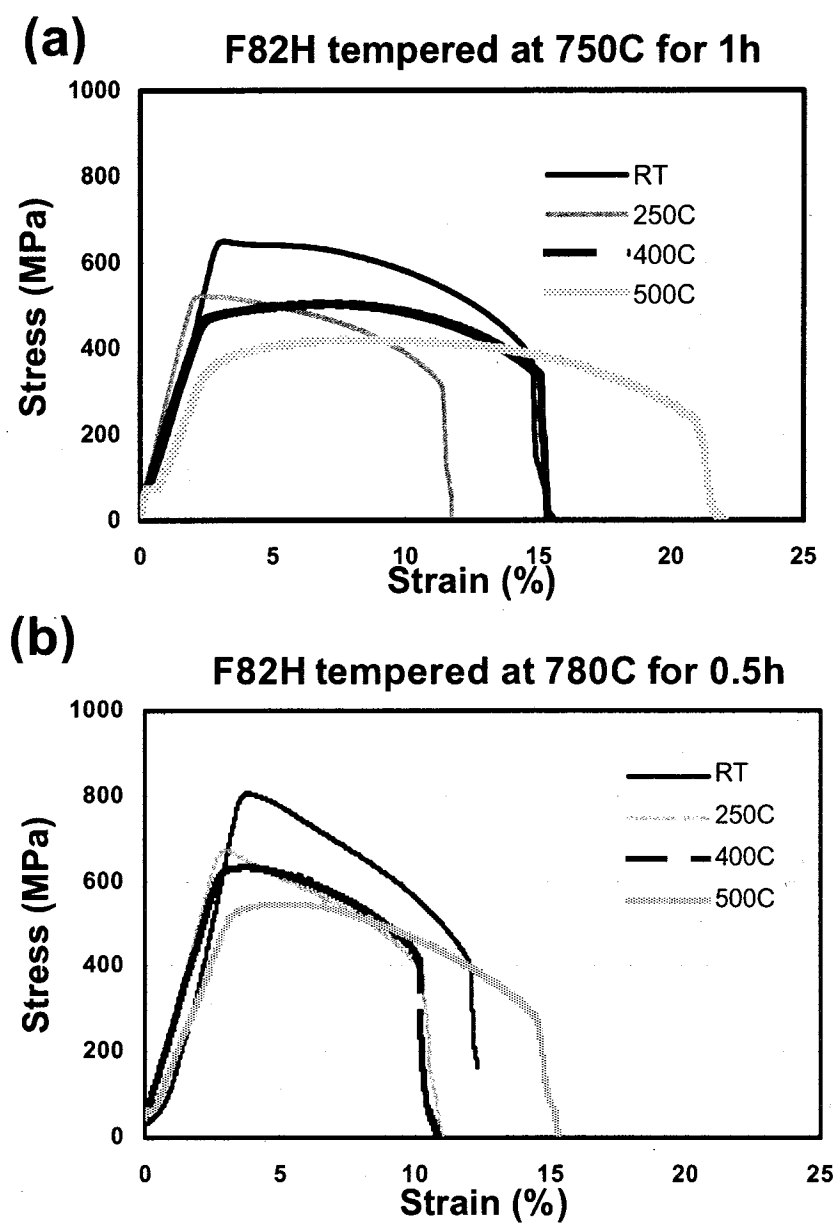


Fig. 5.1 Stress-strain curves of F82H steels irradiated at 250°C to about 2 dpa in JMTR. The specimens were tempered at 750°C for 1 h (a) or 780°C for 0.5 h (b).

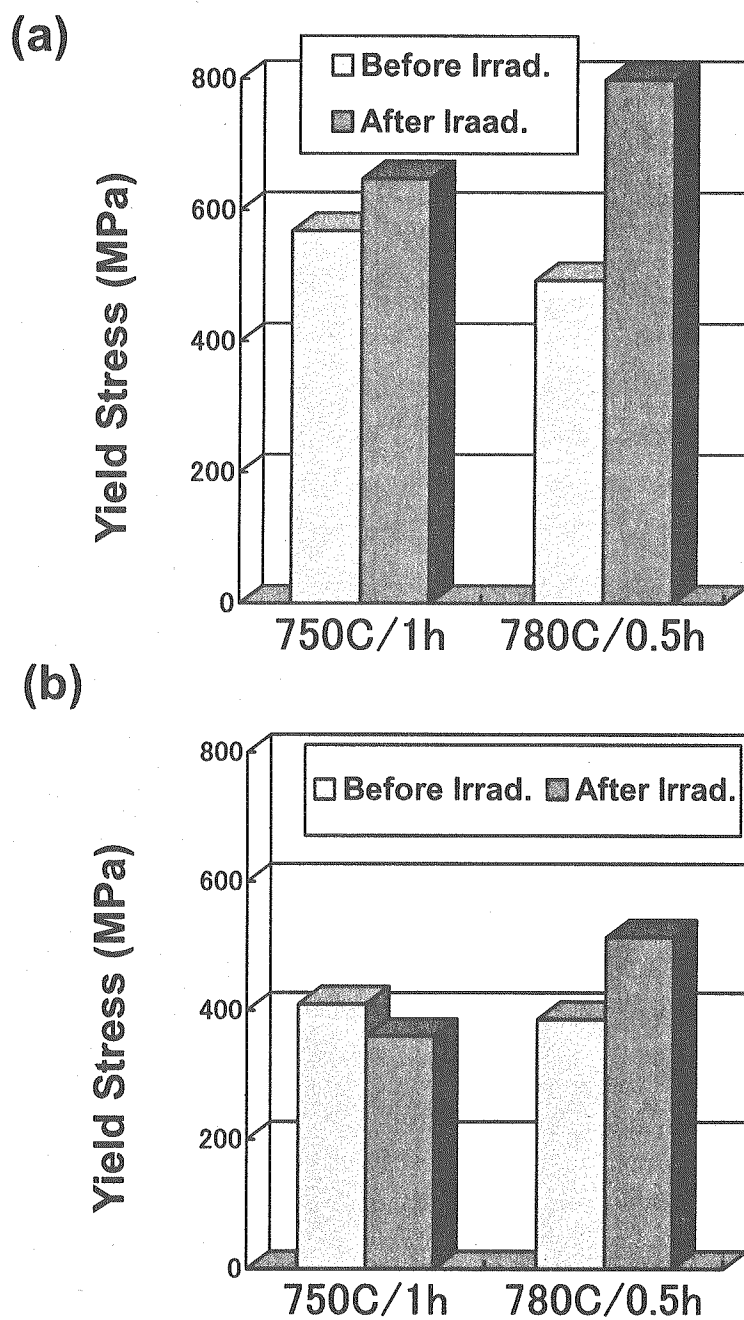


Fig. 5.2 Yield stress of the F82H steels, tempered at 750°C and 780°C, tested at 25 (a) and 500°C (b) before and after the irradiation.

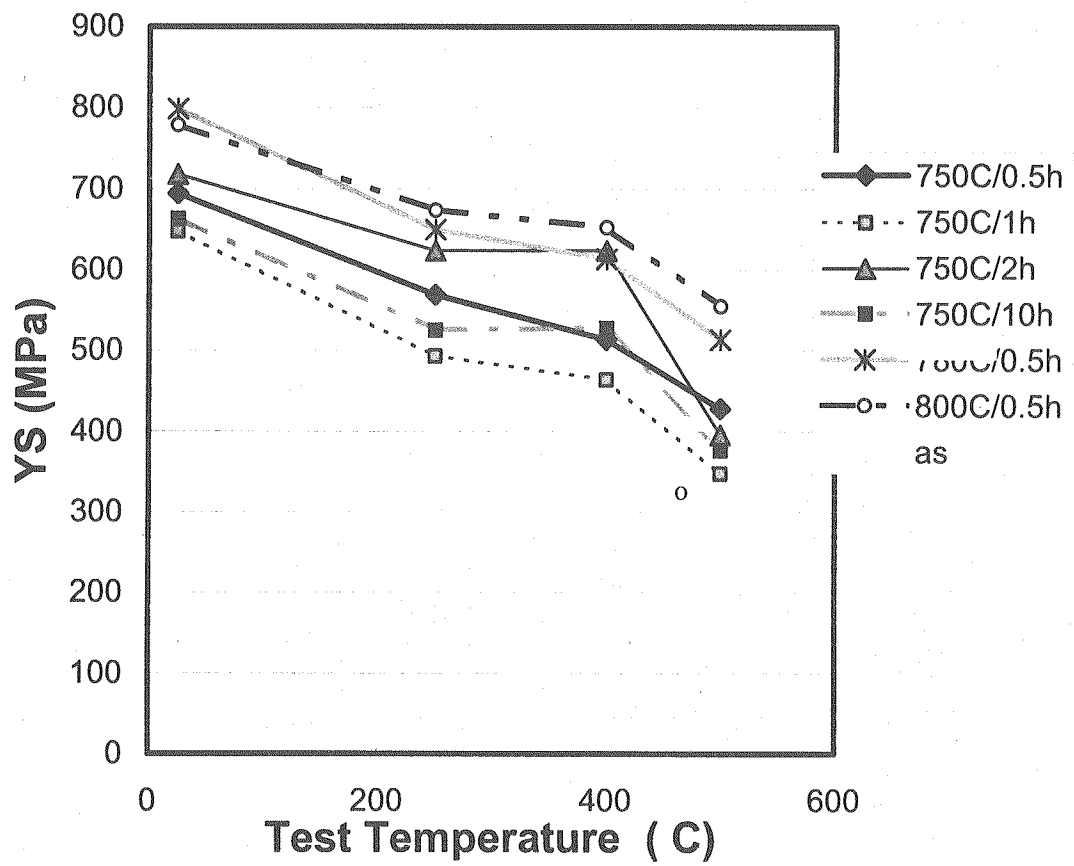


Fig. 5.3 Yield stress measured at 25, 250, 400 and 500°C in the irradiated F82H steel tempered at different conditions.

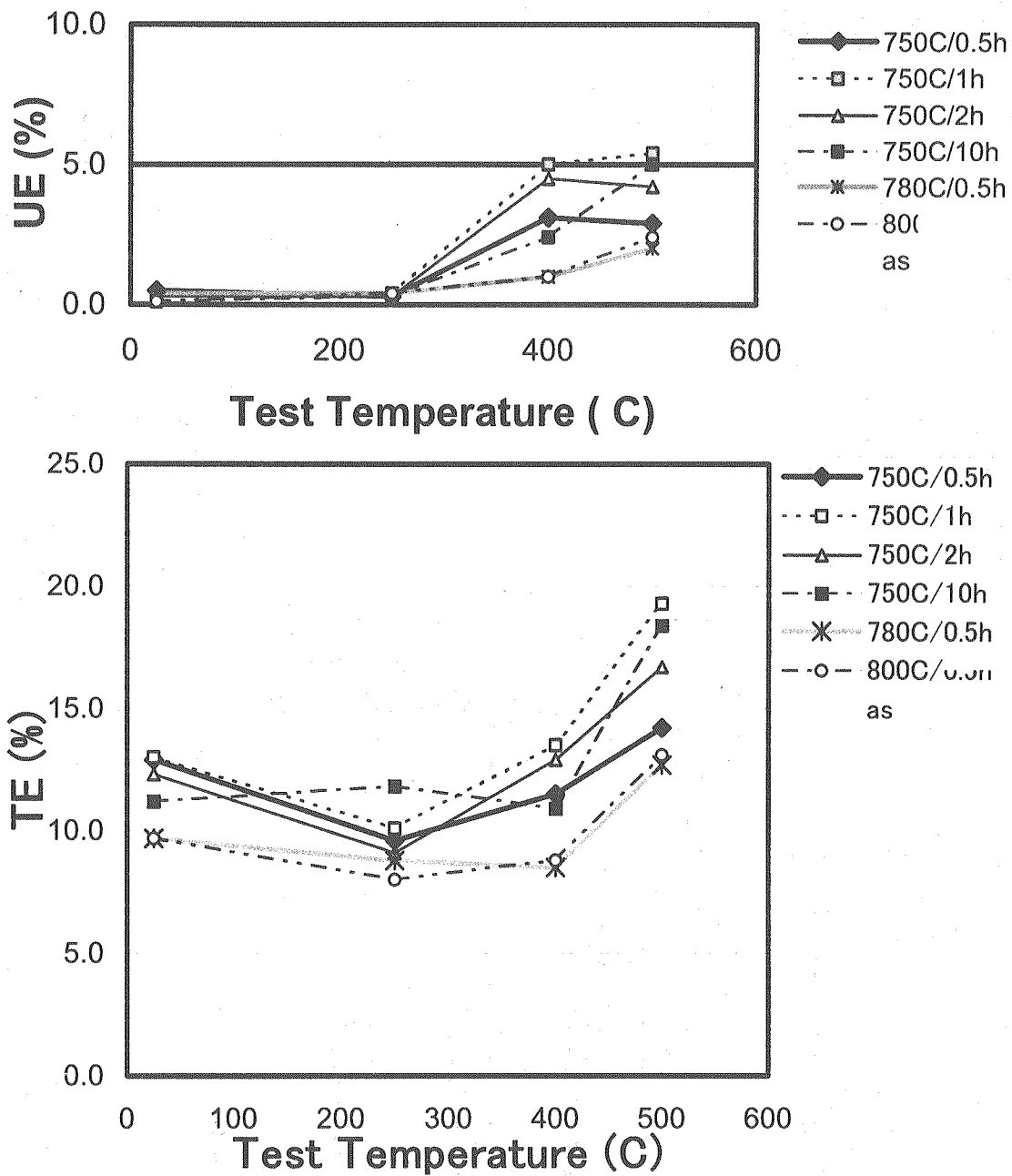


Fig. 5.4 Uniform elongation (a) and total elongation (b) measured at 25, 250, 400 and 500°C in the irradiated F82H steel tempered at different conditions.

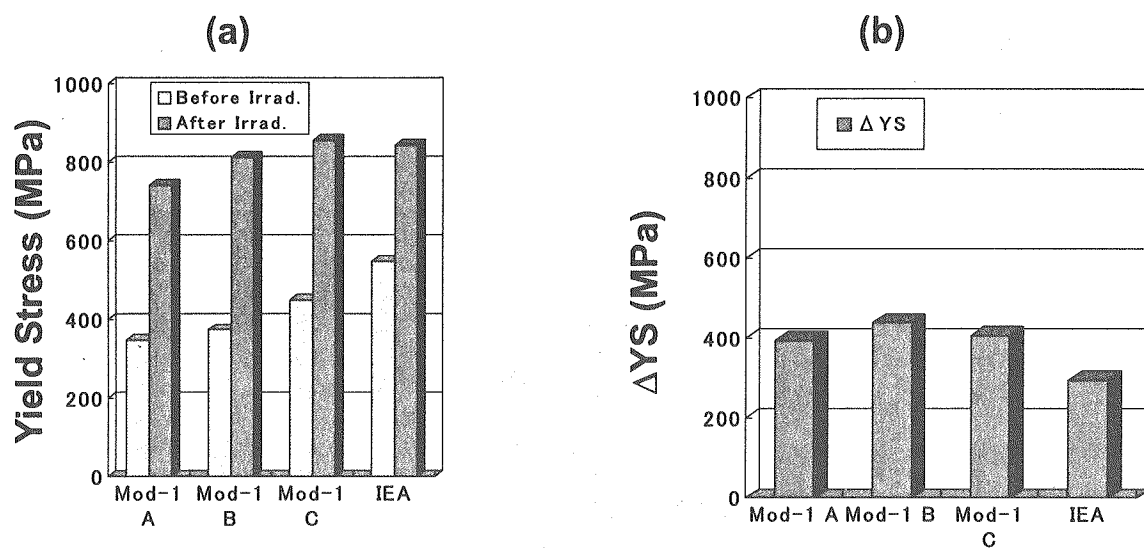


Fig. 5.5 (a) Yield stress and (b) ΔYS of F82H steels tested at 25°C, before and after the HFIR irradiation at 300°C to about 5 dpa.

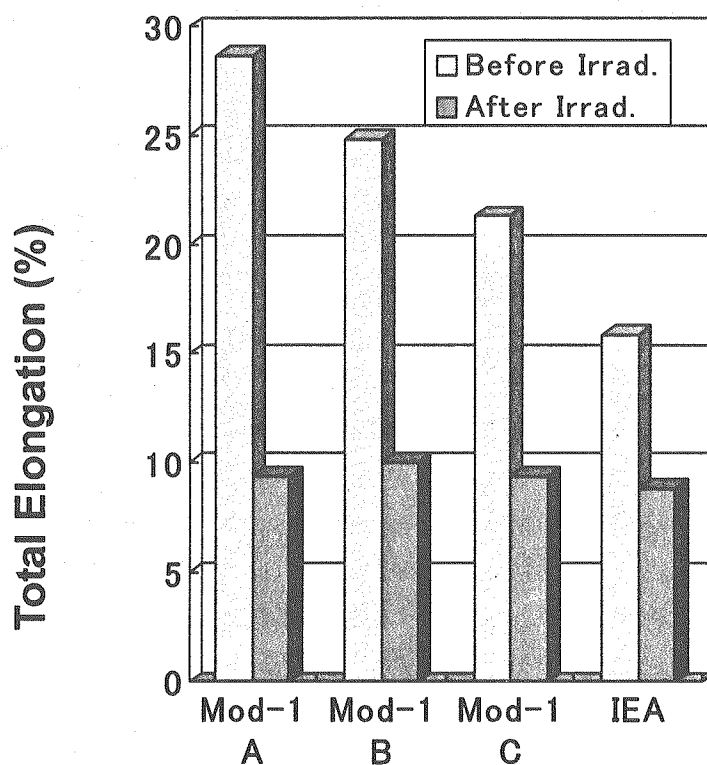


Fig. 5.6 Total elongations of F82H steels tested at 25°C, before and after the HFIR irradiation at 300°C to about 5 dpa.

Table 5.1: Chemical compositions of IEA-heat F82H steel used in this study (wt%)

Element	C	Sol. Al	Si	Mn	P	Si	V	Ti	Cr	Ni
(%)	0.09	0.001	0.07	0.1	0.003	0.001	0.19	0.004	7.82	0.02

Element	Cu	Nb	Ta	W	B	O	N
(%)	0.01	0.002	0.04	1.98	0.0002	-	0.007

Table 5.2: Tensile data of the F82H steels before irradiation

Alloy	Tempering Temp.	Tempering Time	Displacement Damage	Irradiation Temp.	Test Temp.	YS	UTS	UE	TE
	(°C)	(h)	(dpa)	(°C)	(°C)	(MPa)	(MPa)	(%)	(%)
F82H-IEA-Re-N&T	750	0.5	0	-	400	506.0	547.8	1.8	11.2
F82H-IEA-Re-N&T	750	1	0	-	400	470.0	512.4	1.8	10.4
F82H-IEA-Re-N&T	750	2	0	-	400	422	471.6	2.8	13.2
F82H-IEA-Re-N&T	750	10	0	-	400	354	413.6	4.7	15.2
F82H-IEA-Re-N&T	780	0.5	0	-	400	426.0	473.7	2.8	13.1
F82H-IEA-Re-N&T	800	0.5	0	-	400	382.3	444.6	4.1	13.5
F82H-IEA-Re-N&T	750	0.5	0	-	500	451.8	464.8	0.92	14.6
F82H-IEA-Re-N&T	750	1	0	-	500	431.2	457.6	1.3	15.1
F82H-IEA-Re-N&T	750	2	0	-	500	381	405	1.6	15.9
F82H-IEA-Re-N&T	750	10	0	-	500	328	371	3.2	19.1
F82H-IEA-Re-N&T	780	0.5	0	-	500	386.2	415.9	2.0	17.3
F82H-IEA-Re-N&T	800	0.5	0	-	500	354	390	2.4	17.0
F82H-Mod1-A*	700	10	0	-	25	347	497	11.8	28.7
(1)*									
F82H-Mod1-B*	700	10	0	-	25	374	530	11.4	24.8
(1)*									
F82H-Mod1-C*	700	10	0	-	25	449	569	8.7	21.3
(1)*									
F82H-IEA (1)*	750	1	0	-	25	548	652	5.5	15.9

*Heat Conditions: 800C/0.5h+700C/10h (Mod1-A), 860C/0.5h+700C/10h (Mod1-B), 920C/0.5h+700C/10h (Mod1-C)

(1)**Strain rate: 1.1×10^{-3} /s.

Table 5.3: Tensile data of the F82H steels irradiated in JMTR and HFIR

Alloy	Tempering Temp.	Tempering Time	Displacement Damage	Irradiation Temp.	Test Temp.	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
	(°C)	(h)	(dpa)	(°C)	(°C)				
F82H-IEA-Re-N&T	750	0.5	1.9	250	400	513.6	551.9	3.1	11.5
F82H-IEA-Re-N&T	750	1	1.9	250	400	464.1	496.1	5.0	13.5
F82H-IEA-Re-N&T	750	2	1.9	250	400	483.8	515.5	4.5	12.9
F82H-IEA-Re-N&T	750	10	1.9	250	400	528.2	544.2	2.4	10.9
F82H-IEA-Re-N&T	780	0.5	1.9	250	400	613.8	629.7	1.0	8.5
F82H-IEA-Re-N&T	800	0.5	1.9	250	400	652.5	660.4	1.0	8.8
F82H-IEA-Re-N&T	750	0.5	1.9	250	500	428.5	468.2	2.9	14.2
F82H-IEA-Re-N&T	750	1	1.9	250	500	357.7	418.8	5.4	19.3
F82H-IEA-Re-N&T	750	2	1.9	250	500	395.6	450.9	4.2	16.7
F82H-IEA-Re-N&T	750	10	1.9	250	500	377.0	430.8	5.0	18.4
F82H-IEA-Re-N&T	780	0.5	1.9	250	500	513.4	544.5	2.0	12.7
F82H-IEA-Re-N&T	800	0.5	1.9	250	500	554.9	586.6	2.4	13.1
F82H-Mod1-A*	700	10	5	300	25	741	741	0.2	9.4
(1)**									
F82H-Mod1-B*	700	10	5	300	25	812	812	0.2	10.0
(1)**									
F82H-Mod1-C*	700	10	5	300	25	855	857	0.3	9.4
(1)**									
F82H-IEA (1)**	750	1	5	300	25	843	843	0.3	8.9
F82H-Mod1-A*	700	10	5	300	25	697	697	0.2	8.5
(2)**									
F82H-Mod1-B*	700	10	5	300	25	793	797	0.3	9.7
(2)**									
F82H-IEA (2)**	750	1	5	300	25	841	843	0.3	8.9

*Heat Conditions: 800C/0.5h+700C/10h (Mod1-A), 860C/0.5h+700C/10h (Mod1-B), 920C/0.5h+700C/10h (Mod1-C)

(1)**Strain rate: 1.1×10^{-3} /s, (2)**Strain rate: 1.1×10^{-2} /s.

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