



Effect of two-steps heat treatments on irradiation hardening in F82H irradiated at 573 K

M. Ando^{a,*}, H. Tanigawa^a, E. Wakai^a, R.E. Stoller^b

^aJapan Atomic Energy Agency, Tokai, Naka, Ibaraki 319-1195, Japan

^bOak Ridge National Laboratory, Oak Ridge, TN 37831, USA

ABSTRACT

Irradiation hardening and embrittlement due to neutron irradiation around 573 K are the important issues on RAF/M steels. It is expected that the improvement of irradiation hardening might be one of effective ways to control the mechanical properties of RAF/M after irradiation. In this study, the purposes are to investigate the effect of heat treatments on irradiation hardening of irradiated F82H variants and to compare the irradiation hardening based on Δ Hardness with the irradiation hardening obtained by Δ Yield Stress about F82H. Neutron irradiation was performed in HFIR at 573 K. The ion-beam irradiation experiment at \sim 573 K was carried out at the TIARA facility of JAEA. For the results of tensile test and hardness test of F82H and F82H heat treatment variants neutron-irradiated at 573 K, all specimens caused irradiation hardening. The irradiation hardening (Δ Hardness) obtained by hardness test is almost same level for neutron- and ion-irradiated F82H specimens, however irradiation hardening (Δ Yield Stress) of F82H Mod-1 A (two-steps heat treated F82H: high temperature tempering and then low temperature tempering) is smaller than that of F82H.

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1. Introduction

Irradiation hardening and embrittlement due to high-energy neutron irradiation around 623 K are the important issues on reduced-activation ferritic/martensitic steels [1,2]. It is expected that the improvement of irradiation hardening might be one of effective methods to control the mechanical properties of RAF/M after irradiation. Recently, it has been reported that the tempering conditions of F82H strongly affected the void swelling and irradiation hardening behavior in F82H irradiated by ion-irradiation and neutron irradiation [3,4]. Also, it was obtained that the weld joint had less hardening than the base metal from the tensile test results of TIG weldments irradiated in HFIR [5]. These results show that irradiation hardening could be reduced by the optimization of heat treatment condition for F82H.

On the other hands, the hardness test using a pyramidal tip has a potential to be compared very easily by a small volume sample, broken specimens and so on though it is difficult to estimate the material property (e.g. yield stress) from the hardness. If more correlation data between the irradiation hardening and its micro-deformation zone analysis in ion/neutron-irradiated F82H could be obtained, the yield stress may be estimated by micro-hardness test and microstructural observation. In addition, it is important to

estimate the yield stress before/after irradiation to evaluate the model of irradiation hardening, e.g. Orowan-dispersion hardening.

The purposes of this study are to investigate the effect of heat treatments on irradiation hardening of irradiated F82H and to compare the irradiation hardening (Δ Hardness) obtained from Vickers-hardness with the irradiation hardening (Δ Yield Stress) obtained from tensile property about F82H.

2. Experiment

2.1. Materials

The material is F82H IEA heat. The detailed description of the material has been published elsewhere [6]. The heat treatment was normalized at 1313 K for 0.5 h and tempered at temperature of 1023 K for 1 h (F82H Std). In this experiment, five types F82H variants were also prepared by heat treatments in order to compare with the irradiation hardening after irradiation. F82H LT and HT (Tempering condition, LT: 973 K, 1 h; HT: 1073 K, 1 h) were prepared to examine the effect of several tempering temperatures on irradiation hardening. On the other hands, F82H Mod-1 A simulated an over (high temperature) tempered region and F82H Mod-1 B and C simulated a fine grain region in the heat affected zone of weldment. These F82H heat treatment variants were also used to examine the effect of the second lower temperature tempering (973 K) on irradiation hardening. The chemical compositions and these heat treatment conditions are shown in Table 1.

* Corresponding author. Tel.: +81 29 282 6146; fax: +81 29 284 3589.
E-mail address: ando.masami@jaea.go.jp (M. Ando).

Table 1
Chemical composition (mass%) and heat treatment conditions of F82H.

	C	Cr	W	V	Ta	Mn	Si	P	S	N	
F82H IEA	0.09	7.71	1.95	0.16	0.02	0.16	0.11	0.002	0.002	0.006	
	Normalization				First heat treatment		Second heat treatment				
	K		h		K		h		K		h
F82H Std	1313		0.5		1023		1		–		
F82H LT					973		1		–		
F82H HT					1073		1		–		
F82H Mod-1 A	1313		0.5		1073		0.5		973		1–14
F82H Mod-1 B					1133		0.5				
F82H Mod-1 C					1193		0.5				

2.2. Irradiation

Neutron irradiation was performed in the Oak Ridge National Laboratory (ORNL) High Flux Isotope Reactor (HFIR) to atomic displacement levels up to 9 dpa (nominal) in the target position (capsule name: JP26). Nominal irradiation temperature was 573 K. The dimension of SS-J3 sheet type miniature tensile specimen was a gauge section of $7.62 \times 0.76 \times 1.5$ mm. The ion-beam irradiation experiment was carried out at the TIARA facility of JAEA. These specimens were irradiated at 543 and 633 K by 10.5 MeV Fe^{3+} ions. The irradiation dose was performed to 3.5–14 dpa at the depth of 0.6 μm and the damage rate was about 1.0×10^{-3} dpa/s.

2.3. Post-irradiation experiment

The neutron-irradiated specimens were tested at a load of 9.8 N using an AAV-500 micro-Vickers-hardness tester in Hot cell. The hardness test was 8 ~ 10 points for each SS-J3 tensile specimen at the end tab area. Tensile tests on F82H IEA and F82H Mod-1 A, B and C were carried out at a strain rate of $1.6 \times 10^{-3} \text{ s}^{-1}$ at room temperature in Hot Cell Facility, ORNL. The ion-irradiated specimens were tested using an UMIS-2000 (CSIRO, Australia) ultra micro-indentation testing system. All specimens were indented up to ~400 nm from the surface (indentation load 10 ~ 16mN) to compare with micro-hardness easily in before/after irradiation. The micro-indentation results were analyzed in the manner outlined by Oliver and Pharr [7].

3. Results and discussion

3.1. Micro-hardness results for ion-irradiation

In this experiment, the ion-irradiation was performed to examine the effect of heat treatments on irradiation hardening as a pre-neutron irradiation experiment. Fig. 1a shows the relationship between various tempering temperatures and hardness (Vickers-hardness and micro-indentation hardness) for F82H steel before ion-irradiation. The hardness tends to increase as the lower temperature tempering because in the case of low temperature tempered F82H (F82H LT), high-density dislocations are observed in the martensite lath. Therefore, the hardness of F82H LT is higher than that of F82H Std. On the other hand, the hardness of high temperature tempered F82H (F82H HT) is lower than that of F82H Std because dislocations in martensite lath recover at high temperature tempering (e.g. tempering at 1073 K). Fig. 1b shows the micro-hardness in various tempered F82H steels irradiated at 543 K up to 3.5 dpa. The F82H LT caused a large irradiation hardening. Consequently, these results indicate that micro-hardness of irradiated specimens tended to decrease by the higher temperature tempering. Effects of several tempering conditions of F82H on irradiation hardening were also reported by using JMTR irradiation at

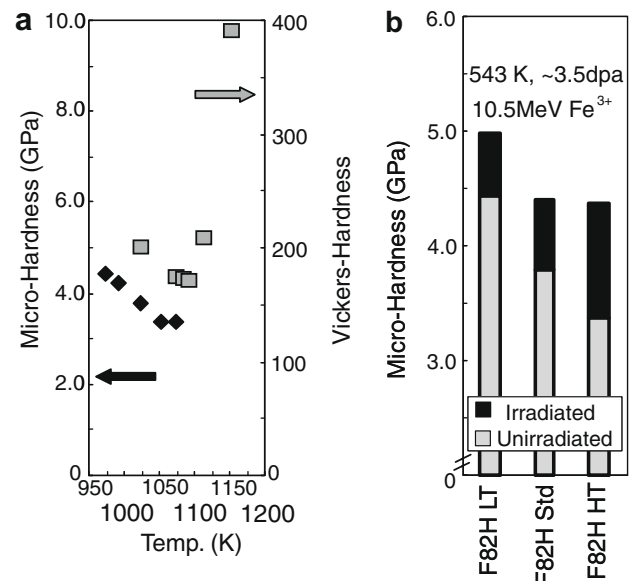


Fig. 1. (a) Relationship between various tempering temperatures and hardness (Vickers-hardness and micro-indentation hardness) for F82H steel. (b) The micro-hardness in various tempered F82H steels irradiated at 543 and 633 K up to 3.5 dpa.

523 K up to 2 dpa [4]. In these results, neutron irradiation hardening of F82H HT (Tempering condition: 1073 K, 0.5 h) also was largest of all specimens. For the effect of two-step heat treatments, F82H Mod-1 variants were irradiated at 543 K for 14 dpa. The results of micro-hardness in these irradiated specimens were shown in Fig. 2a. Irradiation hardening occurred by ion-irradiation at 543 K about F82H IEA and F82H Mod-1 specimens. On the other hand, it is indicated that the micro-hardness of irradiated F82H Mod-1 A is smallest as well as the high temperature tempering specimen.

3.2. Hardness results for neutron irradiation

Fig. 2b shows the relationship between various heat treatment conditions and Vickers-hardness for F82H steels before/after neutron irradiation. Irradiation hardening occurred by neutron irradiation at 573 K about F82H IEA and F82H Mod-1 specimens. This is similar to the result of irradiation hardening in IEA and Mod-1 variants ion-irradiated at 543 K. The lowest hardness is F82H Mod-1 A specimen before/after irradiation because the dislocations in martensite lath recovered under high temperature tempering. Irradiation hardening (the difference between hardness after irradiation and hardness before irradiation) is almost same level.

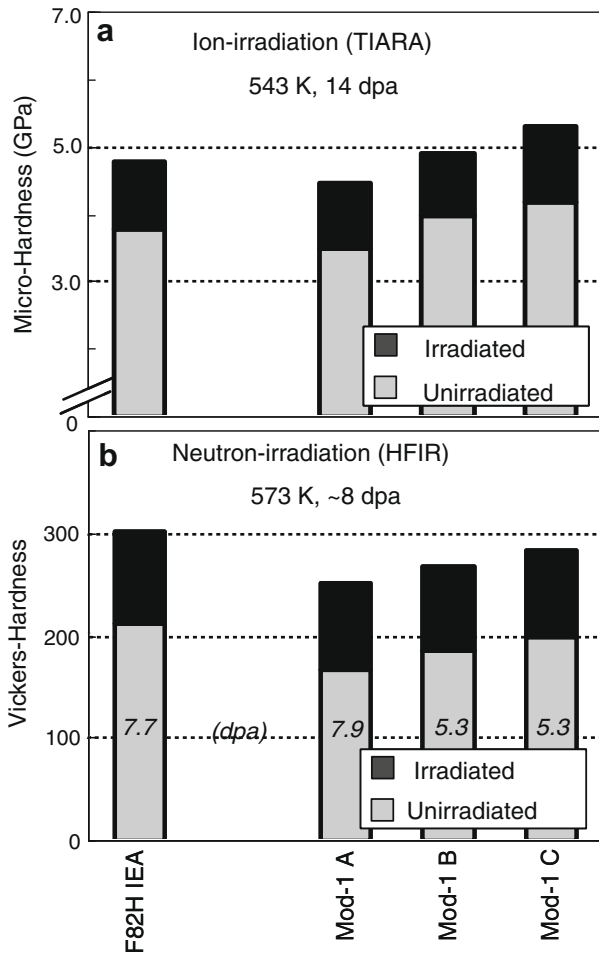


Fig. 2. The results of (a) micro-hardness in the F82H Mod-1 variants ion-irradiated at 543 K for 14 dpa and (b) Vickers-hardness F82H variants neutron-irradiated at 573 K for 8 dpa.

3.3. Tensile properties of JP26 irradiated specimens

The tensile property and hardness obtained from the SS-J3 specimens before/after JP26 irradiation were summarized in Table 2. Increases in yield stress (ΔYS) of F82H IEA and F82H Mod-1 variants after irradiation are shown in Fig. 3 (include RB11J irradiation data). These F82H steels occurred a large irradiation hardening after neutron irradiation at 573 K. The increase of yield stress of Mod-1 C is almost the same as that of F82H IEA. However, the increase of yield stress in Mod-1 A is less than ~30% of that of F82H IEA. These results are similar to the tensile properties of TIG

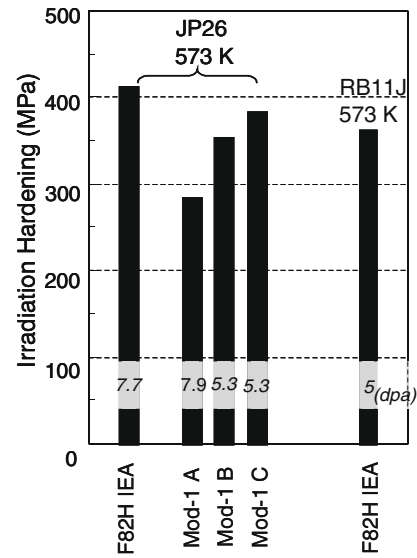


Fig. 3. The irradiation hardening (ΔYS) for F82H IEA and F82H Mod-1 variants irradiated at 573 K.

weldment irradiated at 573 K [5]. This shows that the conditions of heat treatment for smaller irradiation hardening in F82H steel were obtained in this experiment. In this case, it is expected that the first heat treatment (high temperature tempering) caused a reduction of dislocation density, and the second heat treatment (low temperature tempering) contributed to decrease the solute carbon concentration in lath-martensite matrix. Unfortunately, the reduction of irradiation hardening wasn't detected by Vickers-hardness measurement. Moreover, for the results of the ultimate tensile strength (UTS) in irradiated F82H variants, the irradiation hardening behavior is similar to that of yield stress. Relationship between Vickers-hardness (H_v) and UTS (σ_B) is known as below,

$$\sigma_B/H_v=3. \tag{1}$$

In Fig. 4, actual factors which were σ_B/H_v for each specimen in this experiment were plotted. The range of σ_B/H_v was from 2.8 to 3.2.

Also, at 573 K irradiation,

$$\sigma_B=\sigma_y. \tag{2}$$

Therefore, the σ_y (σ_B) of F82H after irradiation at 573 K can be almost estimated by Vickers-hardness.

Detailed examination of relationship between the micro-hardness and the micro-deformation under the indent in irradiated and un-irradiated F82H will be performed to estimate more accurate ΔYS of F82H in future studies.

Table 2
Results of tensile property and hardness for F82H variants before/after neutron irradiation.

Unirradiation	Tensile property				Hardness Hv _{1.0}		
	σ_y (MPa)	σ_B (MPa)	ϵ_u (%)	ϵ_t (%)			
F82H IEA	–	523.0	621.6	5.7	22.4	212	
F82H Mod-1 A	–	434.0	561.0	11.2	31.9	167	
F82H Mod-1 B	–	414.3	567.0	12.8	35.4	185	
F82H Mod-1 C	–	532.0	647.0	7.1	26.6	198	
JP26 irradiation	Irradiation temperature	dpa	σ_y (MPa)	σ_B (MPa)	ϵ_u (%)	ϵ_t (%)	Hv _{1.0}
F82H IEA	573	7.7	935.5	938.9	0.3	11.8	306
F82H Mod-1 A	573	7.9	716.0	723.3	0.5	15.9	252
F82H Mod-1 B	573	5.3	766.5	766.5	0.3	15.6	267
F82H Mod-1 C	573	5.3	914.8	914.8	0.2	12.4	283

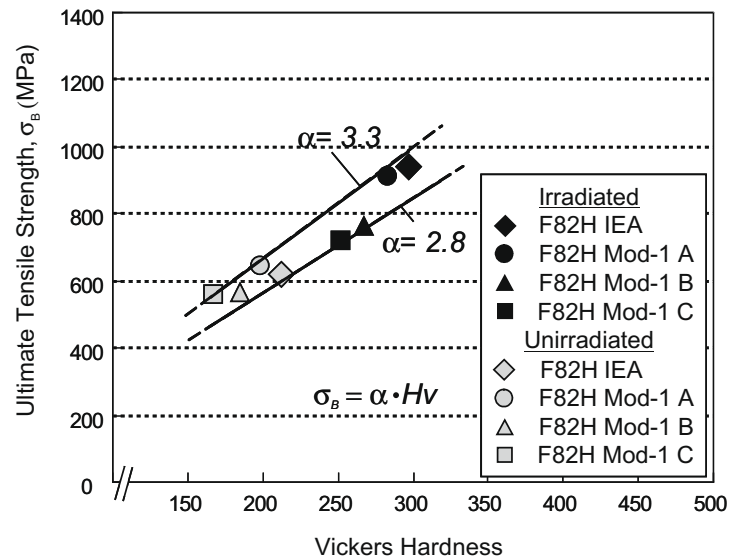


Fig. 4. Relationship between ultimate tensile strength (σ_B) and Vickers-hardness for F82H and F82H heat treatment variants.

4. Summary

- (1) The irradiation hardening (ΔH) obtained by hardness test is almost similar level (neutron- and ion-irradiated F82H variants), however, irradiation hardening (ΔYS) of F82H Mod-1 A is smaller than that of F82H.
- (2) For this result, one of effective methods for reduction of irradiation hardening is two-step heat treatments. It is suggested that the first heat treatment (high temperature tempering) caused a reduction of dislocation density, and second heat treatment (low temperature tempering) contributed to decrease the solute carbon concentration in matrix.
- (3) As results of hardness and ultimate tensile strength (σ_B) of neutron-irradiated SS-J3 type specimens (F82H IEA and Mod-1 A–C), the σ_B (or σ_y) after irradiation at ~ 573 K up to ~ 8 dpa can be estimated by Vickers-hardness results.

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References

- [1] A. Hishinuma, A. Kohyama, R.L. Klueh, D.S. Gelles, W. Dietz, K. Ehrlich, J. Nucl. Mater. 258&263 (1998) 193.
- [2] N. Baluc, D.S. Gelles, S. Jitsukawa, A. Kimura, R.L. Klueh, G.R. Odette, B. van der Schaaf, Jinnan Yu, J. Nucl. Mater. 367&370 (2007) 33.
- [3] E. Wakai, T. Sawai, K. Furuya, A. Naito, T. Aruga, K. Kikuchi, S. Yamashita, S. Ohnuki, S. Yamamoto, H. Naramoto, S. Jitsukawa, J. Nucl. Mater. 307&311 (2002) 278.
- [4] E. Wakai, M. Ando, S. Matsukawa, T. Taguchi, T. Yamamoto, H. Tomita, F. Takada, Fus. Technol. 47 (2005) 856.
- [5] K. Shiba, R.L. Klueh, Y. Miwa, N. Igawa, J.P. Robertson, DOE/ER-0313/28. 131 (2000).
- [6] K. Shiba, A. Hishinuma, A. Tohyama, K. Kasamura, Properties of Low Activation Ferritic Steel F82H IEA Heat, JAERI-Tech 97-038, JAERI, 1997.
- [7] W.D. Oliver, G.M. Pharr, J. Mater. Res. 7 (1992) 1564.