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Mechanical property changes of low activation ferritic/martensitic steels after neutron irradiation

Y. Kohno [a,*], A. Kohyama [b], T. Hirose [b], M.L. Hamilton [c], M. Narui [d]

[a] *Department of Materials Science, The University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113, Japan*
[b] *Institute of Advanced Energy, Kyoto University, Gokasho, Uji, Kyoto 611, Japan*
[c] *Materials Science Division, Pacific Northwest National Laboratory, Richland, WA 99352, USA*
[d] *Institute for Materials Research, Tohoku University, Oarai Branch, Oarai, Ibaraki 311-13, Japan*

Abstract

Mechanical property changes of Fe–*X*Cr–2W–0.2V,Ta (*X*: 2.25–12) low activation ferritic/martensitic steels including Japanese Low Activation Ferritic/martensitic (JLF) steels and F82H after neutron irradiation were investigated with emphasis on Charpy impact property, tensile property and irradiation creep properties. Dose dependence of ductile-to-brittle transition temperature (DBTT) in JLF-1 (9Cr steel) irradiated at 646–700 K increased with irradiation up to 20 dpa and then decreased with further irradiation showing highest DBTT of 260 K at 20 dpa. F82H showed similar dose dependence in DBTT to JLF-1 with higher transition temperature than that of JLF-1 at the same displacement damage. Yield strength in JLF steels and F82H showed similar dose dependence to that of DBTT. Yield strength increased with irradiation up to 15–20 dpa and then decreased to saturate above about 40 dpa. Irradiation hardening in 7–9%Cr steels (JLF-1, JLF-3, F82H) were observed to be smaller than those in steels with 2.25%Cr (JLF-4) or 12%Cr (JLF-5). Dependences of creep strain on applied hoop stress and neutron fluence were measured to be 1.5 and 1, respectively. Temperature dependence of creep coefficient showed a maximum at about 700 K which was caused by irradiation induced void formation or irradiation enhanced creep deformation. Creep coefficient of F82H was larger than those of JLF steels above 750 K. This was considered to be caused by the differences in N and Ta concentration between F82H and JLF steels.

1. Introduction

In order to increase the acceptability of fusion reactors by reducing highly radioactive material generation from the system components, low activation alloy development has been incorporated into the goals of fusion reactor materials development [1]. Of the possible low activation materials, ferritic/martensitic steels are on of the most viable as a first wall structural material for DEMO reactor and beyond. Efforts to develop low activation ferritic/martensitic steels are based on replacement of Mo by W in the conventional Fe–Cr–Mo heat resistant steels [2–7]. A systematic alloy design study was initiated in Japan in mid 1980s in conjunction with the initiation of the Japan/US collaborative irradiation program utilizing the Fast Flux Test Facility/Materials Open Test Assembly (FFTF/MOTA) in Hanford.

Six kinds of Fe–Cr–W based low activation ferritic/martensitic steels have been proposed as leading candidate Japanese Low Activation Ferritic/martensitic steel (JLF) series steels [8,9]. Basic compositions of JLF series steels are Fe–*X*Cr–2W–0.2V–0.07Ta with 0.05N and 0.1C. Chromium concentration varies from 2.25 to 12 mass%. Concentration of alloying elements other than Cr were optimized from the experimental results on several mechanical property inspection programs including high temperature creep, Charpy impact and tensile properties. Small amount of titanium were added to 9%Cr and 12%Cr steels in order to suppress void formation, and also small a amount of nitrogen was

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[*] Corresponding author. Tel.: +81-3 3812 2111/7128; fax: +81-3 3815 8363; e-mail: kohno@steel.mm.t.u-tokyo.ac.jp

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added for high temperature mechanical property improvement [10].

The objective of this paper is to review the results of mechanical property examinations in JLF series steels after heavy neutron irradiation utilizing the FFTF/MOTA, and to give an input to the material selection for the structural material of future fusion system.

2. Experimental Procedure

2.1. JLF steels

Table 1 shows the chemical composition and heat treatment condition of the developed JLF series steels together with JAERI's F82H and other fusion relevant ferritic/martensitic steels. Baseline composition of JLF series steels is Fe–(2.25–12)Cr–2W. Although the composition optimization concept is described in detail elsewhere [9–12], a brief summary of concepts is as follows. The addition of W instead of Mo improves creep rupture strength in high Cr steels, but excess additions, more than 2–3%, causes toughness degradation due to δ-ferrite formation. Optimum W concentration is considered to be 2% in this class of high Cr steels. V additions also improve high temperature creep rupture strength. The optimum V concentration is determined to be around 0.2%. Excess V addition causes significant coarsening of carbides at high temperature which leads to severe degradation of mechanical properties. Nb has a similar role as V, but it is harmful element from the standpoint of low activation alloy design. Because Ta has equivalent role to Nb, Ta was chosen to be added to JLF series steels for high temperature strength improvement. Optimum Ta concentration was decided to be 0.07–0.08%. Although the effects of Ti addition on mechanical properties of ferritic steels of concern have not been defined clearly, a small amount of Ti (0.015%) was added to 9%Cr and 12%Cr steels to suppress irradiation induced void formation as in the case of austenitic steels. N is considered to be less desirable element from low activation composition. However, a small amount of N was added expecting an improvement of high temperature creep strength.

2.2. FFTF/MOTA irradiation

Miniaturized mechanical property test specimens such as tensile, Charpy, creep (pressurized tube type) and disk bend tests as well as TEM microstructural disk specimens of JLF series steels were included in the MOTA canisters of FTR (Fast Test Reactor) in the FFTF. Neutron irradiation was performed in FFTF cycle-11 (MOTA-2A) and/or cycle-12 (MOTA-2B) at the temperature range 663–873 K. Accumulated fast neutron fluences were 8.6×10^{22} (MOTA-2A, 35 dpa), 5.8×10^{22} (MOTA-2B, 25 dpa) and 1.4×10^{23} (throughout MOTA-2A and 2B, 60 dpa) n/cm^2. Tensile tests were carried out at irradiation temperature utilizing the automated tensile testing machine MATRON which was specially developed for the FFTF/MOTA irradiation program. Tensile specimens used were miniaturized specimen (S-size) with the dimension of 16 mm L × 4 mm W × 0.25 mm T with gauge part of 5 mm L × 1.2 mm W. Disk bend tests was carried out by means of the 3-point bend method using 3 mm diameter TEM disks jointly with tensile tests in order to estimate irradiation hardening of materials. Miniaturized Charpy impact tests using 1.5 mm size small specimens (1.5 mm × 1.5 mm × 20 mm, V-notch: 30° × 0.2 mm depth) were also performed utilizing a specially developed Charpy machine, in the test temperature range from 75 to 373 K [13]. Irradiation creep measurements were performed by means of pressurized tube method using small tube specimens filled by high purity Ar gas. Maximum hoop stress in the tube wall was 240 MPa. Irradiation creep was estimated through measuring diametral dimension change of individual creep tube by the precision laser ruler after each irradiation.

Table 1
The chemical composition and the heat treatment condition of JLF series steels

	C	Mn	Si	Cr	Mo	Ti	W	Ta	V
JLF-1	0.10	0.46	<0.1	9.04	<0.01	0.001	1.97	0.07	0.19
JLF-2	0.10	0.45	<0.1	9.16	<0.01	0.010	1.93	0.07	0.20
JLF-3	0.09	0.45	<0.1	7.03	<0.01	0.001	1.97	0.07	0.20
JLF-4	0.10	0.50	<0.1	2.23	<0.01	0.003	1.97	0.07	0.20
JLF-5	0.09	0.48	<0.1	11.99	<0.01	0.002	1.98	0.07	0.19
JLF-6	0.10	0.46	<0.1	12.00	<0.01	0.010	1.94	0.07	0.19
F82H	0.09	0.49	<0.1	7.65	<0.01	–	2.00	0.04	0.18
HT-9	0.20	0.50	0.50	11.00	1.00	0.5Ni	0.50	–	0.30

Heat treatment: JLF-1,2,3,5,6: 1323 K × 1.8 K s (normalizing) + 1048 K × 3.6 K s (tempering); JLF-4: 1323 K × 1.8 K s (normalizing) + 973 K × 3.6 K s (tempering).

3. Results

3.1. Irradiation hardening of JLF series steels

Fig. 1 shows the 3-point disk bend test results of JLF series steels, F82H and HT-9 following irradiation to 35 dpa, showing the irradiation temperature dependence of flexural yield strength. Yield strength of 7–9%Cr steels (JLF-3, F82H, JLF-1) following irradiation are less temperature dependent than 2.25%Cr (JLF-4) and 12%Cr (JLF-5, HT-9) steels, and are much lower than those of JLF-4 and JLF-5 at irradiation temperatures below 720 K. It is seen that irradiation hardening occurred at temperatures below about 720 K and softening above that temperature. Hardening in 7–9%Cr steels is much smaller than that in 2.25%Cr and 12%Cr steels, and especially in 8%Cr steel (F82H), irradiation hardening was measured to be negligibly small. Low Cr steel (2.25%Cr, JLF-4) showed significant softening at higher irradiation temperatures. Fig. 2 shows temperature dependence of yield strength obtained by tensile test from MATRON following irradiation to 35 dpa. Although high temperature data is not available at present, tensile results seem to be essentially the same as disk bend results, indicating the same material and temperature dependence of yield strength. Fig. 3 shows the dose dependence of tensile yield strength of steels irradiated and tested at 656–700 K. Yield strength once increases with irradiation dose to about 15–20 dpa, and then decreases through irradiation to 35 dpa followed by the saturation over 35 or 40 dpa. It is likely that there might be a peak in yield strength at around 20 dpa. Fig. 4 summarizes the material and irradiation temperature dependence of tensile yield strength from all irradiation conditions. It is clearly seen that steels in concern are assorted to two classes: 7–9%Cr steels and (2.25%Cr, 2%Cr) steels.

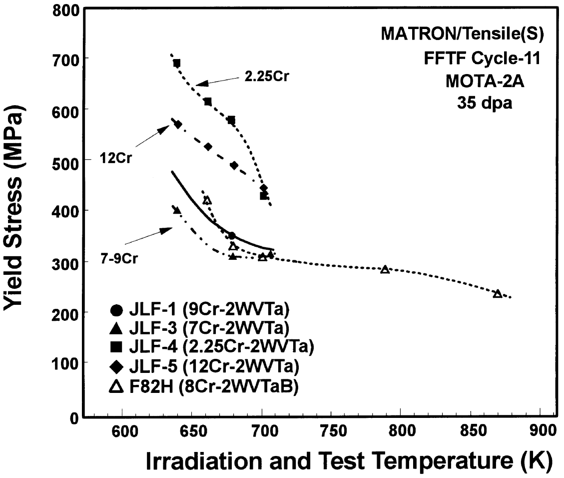

Fig. 2. Temperature dependence of tensile yield strength following irradiation to 35 dpa in the FFTF/MOTA.

3.2. Irradiation creep

Creep deformation of ferritic steels under irradiation condition is one of the most important issues to be clarified for design criteria definition of future fusion reactor core structures. It is quite difficult to perform in-pile and in situ measurement of creep deformation, so that pressurized tube method has been widely utilized to measure irradiation creep in fusion materials irradiation experiments as in this FFTF/MOTA irradiation experiment. Pressurized tube creep results have been formulated qualitatively in the equation: $\varepsilon = B(\phi t)\sigma^{1.5}$, where ε

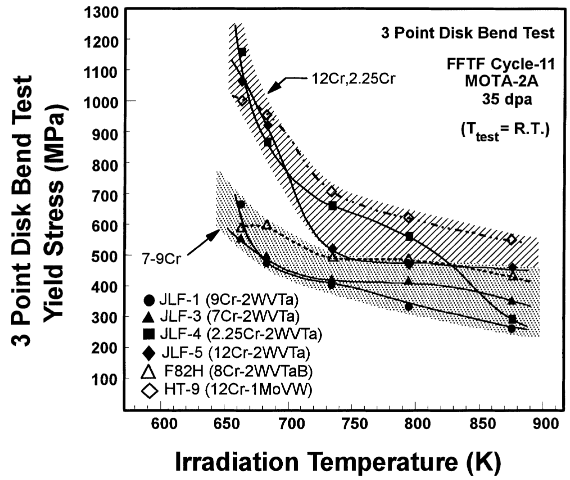

Fig. 1. Temperature dependence of flexural yield strength following irradiation to 35 dpa in the FFTF/MOTA.

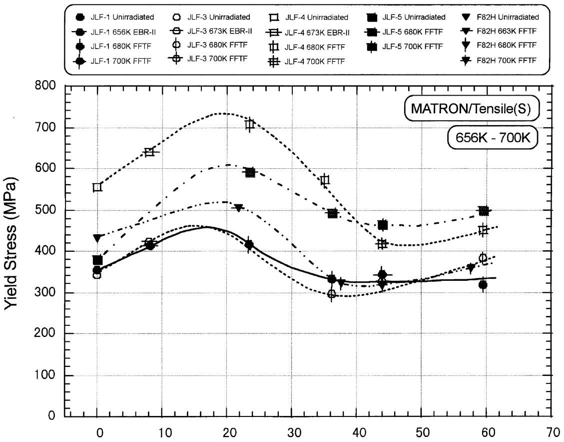

Fig. 3. Dose dependence of tensile yield strength following irradiation at 656–700 K in the FFTF/MOTA or EBR-II.

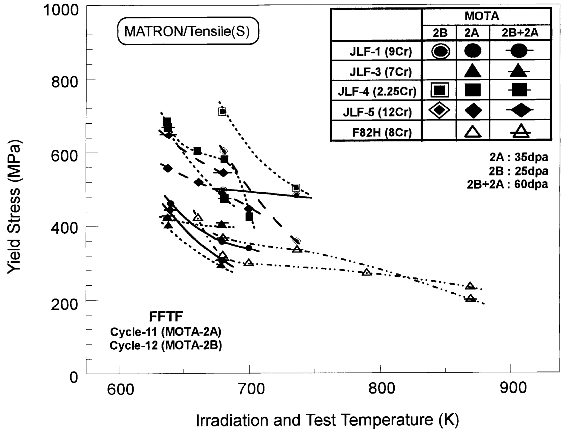

Fig. 4. Irradiation temperature and dose dependences of tensile yield strength of JLF steels and F82H irradiated in the FFTF/MOTA.

is effective creep strain, ϕt expresses irradiation dose, σ is applied stress, and B is average creep coefficient. In Fig. 5, creep measurement results are summarized as irradiation temperature dependence of average creep coefficient. Because average creep coefficient expresses resistant strength to creep deformation, 2.25%Cr steel (JLF-4) has an excellent creep deformation resistance in the steels in concern, though thermal creep components are included in B in Fig. 5. Average creep coefficient B does not show monotonous temperature dependence as expected from thermal creep deformation, but indicates very complicated temperature dependence such as existence of an peak at lower temperature range. 7–9%Cr steels seem to be less creep resistant than other steels, and in F82H (8%Cr), creep resistance is very poor at higher temperature.

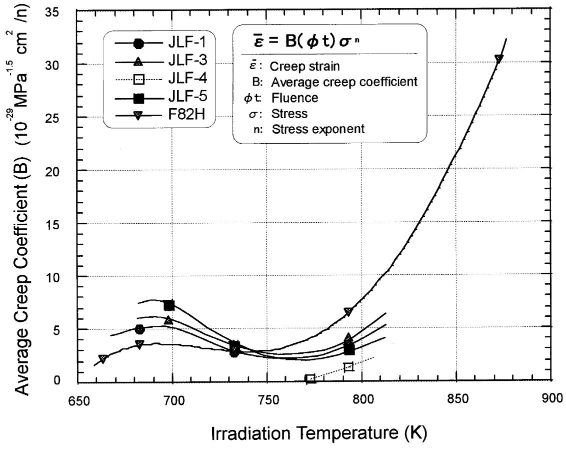

Fig. 5. Temperature dependence of average creep coefficient in JLF steels and F82H.

3.3. Irradiation embrittlement

Irradiation embrittlement is the most important problem of ferritic/martensitic steels in case of fusion application. In this experiment, irradiation embrittlements of steels were evaluated by means of miniaturized Charpy impact tests using 1.5 mm size specimens. Although examinations are not completed yet at present, interesting results have been obtained on 9%Cr steel (JLF-1) and 8%Cr steel (F82H). Fig. 6 shows the Charpy impact curves of JLF-1 after irradiation up to 60 dpa at 646–700 K. Ductile-to-brittle transition temperature (DBTT) of JLF-1 increases after irradiation to 35 dpa, and then decreases by further irradiation to 60 dpa. Although data from 25 dpa irradiation are not obtained, it is likely that the highest DBTT in JLF-1 lies around 215 K. Degradation of toughness (decrease of upper shelf energy) is not so large in comparison with other steels such as HT-9 [14], and is observed to be less than 20%. DBTT in F82H irradiated to 35 dpa lies around 250 K which is 35 K higher than that in JLF-1 of the same irradiation. There are not enough data to discuss the effects of irradiation on dose dependence or materials dependence of DBTT and toughness of steels in concern, however, it is possible to define that DBTT of 7–9%Cr steels will be suppressed to be below room temperature even after heavy irradiation of 60 dpa given in this experiment.

4. Discussion

It has been widely established in this decade that the low activation ferritic/martensitic steel should exclusively be a candidate structural material for the future fusion system, however, material optimization or selection for a leading candidate steel is still under way. The R&D of JLF series steels in Japanese universities' program is a part of the effort to select a leading candidate

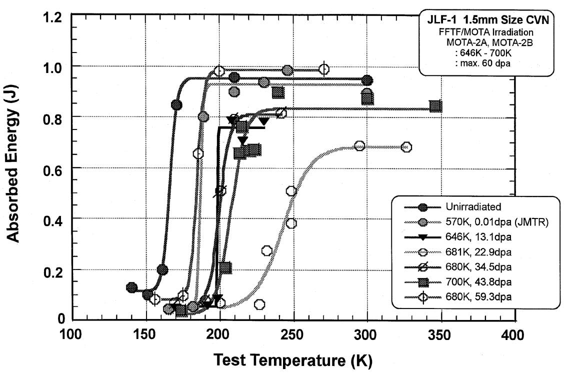

Fig. 6. Charpy impact curves for JLF-1 following irradiation up to 60 dpa.

ferritic/martensitic steel. TEM microstructural examinations of irradiated JLF series steels have characterized the irradiation response in microstructure of JLF series steels, and have shown satisfactory microstructural stability especially in 9%Cr steel (JLF-1) and F82H [15–17]. Precipitation analyses of JLF-1 and F82H indicated that no irradiation induced precipitation was produced which might be at the origin of irradiation hardening. This is one of the reasons for the small or negligible irradiation hardening in 7–9%Cr steels as shown in Fig. 1. This might be also the reason why irradiation embrittlement in JLF-1 and F82H are quite suppressed compared with other classes of steels (Fig. 7). In 12%Cr steel (JLF-5), a high density of very fine precipitates were found by TEM, which were considered to be α' phase. This could be responsible for large irradiation hardening in JLF-5.

Temperature dependence of average creep coefficient was very complicated as shown in Fig. 5. Peak creep temperature was observed at around 425 K. From the results of TEM microstructural observation, each steel shows void formation at this temperature range. Although it is necessary to quantify it properly, peak creep phenomenon at around 425 K might be caused by the void-swelling enhanced creep deformation. In F82H, creep deformation resistance was seen to be very poor at high temperatures. This is because F82H has not a fully martensitic matrix structure, but has a fair amount of bainitic structure which is less stable at high temperature.

From the results of overall mechanical property examinations and microstructural inspections together with precipitation analyses, it is clearly defined that, in ferritic/martensitic steels, stable precipitation response as well as stable fully martensitic matrix structure during irradiation are essential factors to keep good mechanical properties after irradiation. In this sense, 9%Cr steel (JLF-1) has the best performance under irradiation because of stable microstructural and precipitation response, resulting in superior mechanical properties during irradiation.

5. Conclusions

1. R&D program of low activation ferritic/martensitic steel in Japanese universities proposed 6 kinds of Fe–Cr–W candidate steels (JLF series steels) with 2.25–12%Cr and 2%W.
2. Mechanical property examinations of JLF series steels and JAERI's F82H after heavy neutron irradiation by means of FFTF/MOTA provided;
   - ○ Three point disk bend test and tensile test results following irradiation to 35 dpa showed serious hardening in 2.25%Cr and 12%Cr steels below 720 K, while in 7–9%Cr steels, hardening was small or negligible.
   - ○ Dose dependence of yield strength after 683 K irradiation seems to show peak hardening dose at around 10 or 15 dpa followed by decrease and saturation over 35 or 40 dpa. Saturated yield strength in 7–9%Cr steels are lower than other steels. Dose dependence of uniform and total elongation also seemed to saturate following irradiation over 35 or 40 dpa, and uniform or total elongation in 7–9%Cr steels are relatively larger than in 2.25%Cr or 12%Cr steel.
   - ○ Average creep deformation coefficient shows complicated temperature dependence with a peak creep temperature at around 700 K which is likely to be caused by void-swelling enhanced deformation.
   - ○ DBTT in 9%Cr steel (JLF-1) or 8%Cr steel (F82H) still remains below room temperature after heavy neutron irradiation of 60 dpa. DBTT in JLF-1 shifts to lower temperature after irradiation over 35 dpa.
3. Overall mechanical property test results and microstructural evidences give the direction that 9%Cr JLF-1 is the best candidate low activation ferritic steel as future fusion structural material.

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