Progress of Reduced Activation Ferritic/Martensitic Steel development in Japan

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Abstract. Recent accomplishment by the Japanese activity for the reduced activation ferritic/martensitic steel (RAF/M) development has been reviewed. Some of the results obtained in EU and US by international collaborative activities are also introduced. Effect of irradiation on the shift of ductile-to-brittle transition temperature (DBTT) has been evaluated to a dose of 20dpa. Results suggest that RAF/M appears to satisfy the requirement on DBTT-shift for the blanket application in the dose range up to several tens of dpa. Also, enhancement effect of DBTT-shift by transmutation produced helium (He) atoms was revealed to be smaller than has been suggested previously. Preliminary studies about the effect of irradiation on fatigue mechanism, the susceptibility to environmentally assisted cracking in water and flow stress-strain relation have been conducted for the specimens irradiated to several dpa, including the post irradiation tensile property examination of the joints by Hot-isostatic press (HIP) bonding method. The results also indicate that RAF/Ms exhibit suitable properties for ITER test blanket module.

1. Introduction

Development of structural materials with high enough resistance for irradiation damage by fusion neutrons and the verification of blanket designing technology by the operation of ITER test blanket module (ITER TBM) are necessary to construct the blanket for the fusion power demonstration plant (DEMO blanket) [1,2]. For the designing and licensing of ITER TBM, design criteria compatible with the irradiation induced property change of the materials need to be prepared. Because of the good industrial bases and the superior resistance for irradiation, reduced activation ferritic/martensitic (RAF/M) steel is recognized to be the leading candidate structural material for ITER TBM, as well as that for DEMO blanket [3]. Issues for the applications of RAF/M steels to DEMO blanket and ITER TBM are summarized in table1.

Table 1 Issues for the application of RAF/M alloys to DEMO blanket and ITER TBM

- Alloy development for DEMO blanket (Damage levels; >100dpa and >1000appmHe)
    - Short-term mechanical properties up to high damage levels
        - Tensile property
        - Fracture toughness (including, He effect on DBTT shift)
    - Reduced activation steel making
    - High temperature strength (improvement by ODS technique)
- Application to ITER TBM (Damage levels; >3dpa and >30appmHe)
    - (Short-term mechanical properties)
    - Fatigue property
    - Creep property
    - Compatibility (including, environmentally assisted cracking)
    - Materials engineering and Design methodology
        - Joining (mainly, TIG[1] welding and HIP joining)
        - Effect of irradiation condition change (eg. Enhanced hardening)
        - Margin to fracture

[1] Tungsten inert gas arc welding.

In this paper, the progress accomplished during last two years on Japanese RAF/M steels about the evaluation of short-term mechanical properties, fatigue properties, compatibility with water, effect of irradiation temperature change and margin to ductile fracture are introduced, as well as the development of oxide dispersion strengthened (ODS) RAF/M steels for the high temperature application [4]. For the evaluation of irradiation effects, fission reactors and multi-ion beam irradiation facilities have been used. Most results for the mechanical properties do not include the effect of transmutation produced He atoms. For the development of RAF/M steels, international collaborations, such as the collaboration of IEA fusion materials development and Japan-U.S. collaborations for fusion reactor materials development (eg. JAERI-DOE HFIR collaboration), are playing vital role. The results obtained by those activities are also introduced.

2. Reduced Activation Ferritic/Martensitic Steels

Progresses about the two alloys developed in Japan are introduced in this paper. The alloys are JLF1 and F82H. Chemical compositions and examples of heat treatment conditions are listed in table 2 [5,6]. JLF1 was developed by Japanese universities, while F82H was developed by the Japan Atomic Energy research Institute and JFE corporation (former, known as NKK). The microstructures of both of these alloys are of tempered martensite. Chromium levels were chosen to minimize irradiation hardening for both alloys [7]. Especially for F82H, chemical composition was selected to keep the unirradiated mechanical properties within similar levels of those of commercial 9Cr-1MoNbV alloys without changing heat treatment specifications. One of the advantages of this strategy of making minor changes to a code-approved material is that it may result in an easier and more rapid qualification of the new alloy. This may also encourage the use of the alloy in the core components, such as TBM of the fusion experimental reactor ITER. Difference between the properties of JLF1 and those of F82H was not large, including those after irradiation. Therefore, results of F82H are mainly introduced in the following.

Table 2 Chemical Composition of Japanese Reduced Activation Martensitic Steels

	C	Si	Mn	Cr	V	W	N	Ta
F82H[1]	0.09	0.10	0.21	7.46	0.15	1.96	0.006	0.023
JLF-1	0.1	0.2	0.45	9.0	0.25	2.0	0.05	0.07

[1] IEA-F82H (IEA heat of F82H, verified under IEA collaboration for Fusion materials development)

Heat Treatment F82H: Normalization;1040C for 38min, Tempering; 750C for 1h
JLF-1: Normalization;1050C for 1h, Tempering; 780C for 1h

3. Tensile Properties

Figure 1(a) shows engineering stress strain curves of JLF1 before and after irradiation in HFIR to 4.8dpa [4]. Hardening and reduction of elongation occurred by irradiation at 350C. Damage level dependence of yield stress for F82H irradiated in HFR and HFIR are shown in Fig. 1(b) [8]. Yield stress linearly increased with logarithm of the damage level to 10-20 dpa at temperatures below 300C, while little hardening occurred at temperatures above 400C. Irradiation to higher damage levels in a fast breeder reactor on 9-12%Cr containing martensitic steels and by an ion beam irradiation facility on F82H exhibited that irradiation hardening saturates with dose at about 10-20dpa [8,9].

4. Fracture Toughness Properties

Hardening by irradiation at temperatures below 400C reduces fracture toughness, including DBTT-shift to higher temperatures [4,8,10,11]. Figure 2 shows damage level dependence of DBTT irradiated at temperatures between 300 and 400C. DBTT was obtained from the results of impact tests with small notched bend bar (miniaturized Charpy V-notch; MCVN) specimens of 3.3mm-cross section and 25mm-long or those with slightly larger sizes (KLST specimens). The DBTT shift tends to saturate with dose, as seen in the figure. Comparing with the results of non-reduced activation alloy (MANET I and II), RAF/Ms exhibited smaller shift. This is suitable for application to blanket.

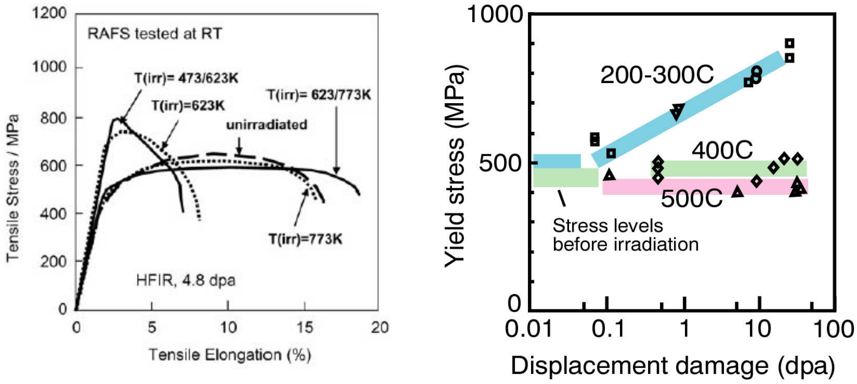


Fig. 1 Tensile properties of JLF-1 and F82H after irradiation
(a) Engineering stress-strain relation of irradiated JLF-1 and (b) Damage level dependence of Yield stress of F82H. Irradiation was conducted in the HFIR.

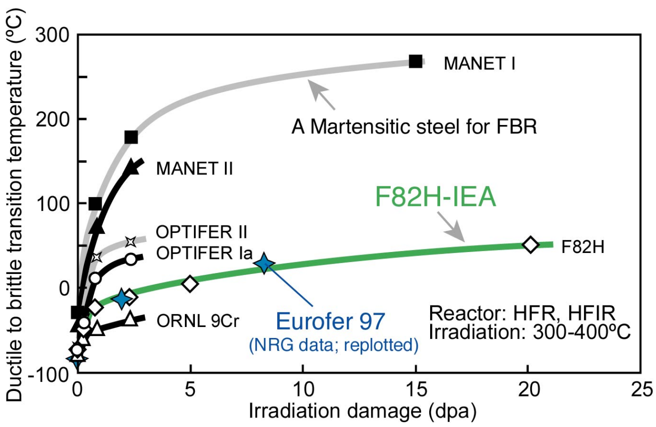
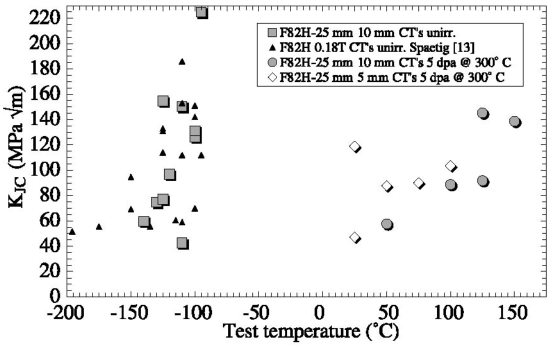


Fig. 2 Damage level dependence of DBTT-shift of several RAF/M steels (Optifer, F82H and ORNL 9Cr)

It is desirable that the DBTT does not exceed 100C for a water-cooled blanket application [8]. Although DBTT is readily affected by specimen size, F82H appears to satisfy the requirement in the dose range up to several tens of dpa for the impact tests with MCVN and KLST specimens. However, the results exhibit the effect of displacement damage only, and transmutation produced He atoms have been suggested to enhance the DBTT-shift [12,13,14]. Figure 3 shows the temperature dependence of the fracture toughness of F82H after irradiation at 300C to about 2dpa [11]. Disk shaped specimens of 12.5 mm-diameter and 4.5 mm-thickness were used for the test. DBTT defined by the fracture toughness value of 100MPam$^{1/2}$ after irradiation are close to 100C. Results are suggesting the specimen size effect. Therefore, care should be taken for the minimum dimension of the component used to examine the degradation of the fracture toughness [8,15,16].
Fusion neutron irradiation introduces appreciable level of transmutation produced He atoms in the material. It has been suggested that He atoms of 100appm fraction caused to enhance

DBTT-shift by more than 100C. The cross section to produce He atoms for fission neutron irradiation is quite small. Therefore, B and Ni are often doped in RAF/Ms to produce He atoms during irradiation in fission reactors. Because B and Ni affect the irradiation induced property changes other than introducing He atoms, the results by fission reactor irradiation need to be analyzed to extract the He effect. DBTT-shift results are summarized against hardening by irradiation in Fig. 4. DBTT-shift seems to be accompanied with irradiation hardening such that ΔDBTT (in C) = $A\Delta\sigma_{ys}$ (in MPa), where the proportional constant A is in the range of 0.47+/-0.19 (C/MPa) [8]. The changes by doping of B and Ni are indicated by dashed arrows in the figure. It seems that the additional hardening by doping mainly causes the additional DBTT-shift. The gradient of the dashed arrows may indicate the He effect on DBTT-shift. The increased effect of He seems to be about one third or even smaller than has been indicated previously. Moreover, the limited amount of He level during ITER TBM application does not seem to introduce a serious problem.

Fig. 3 Temperature dependence of fracture toughness after irradiation to 2 dpa at 300C in HFIR (right) [11].
Disk Compact Tension specimens of 12.5 mm-diameter and 4.5 mm-thickness were used.



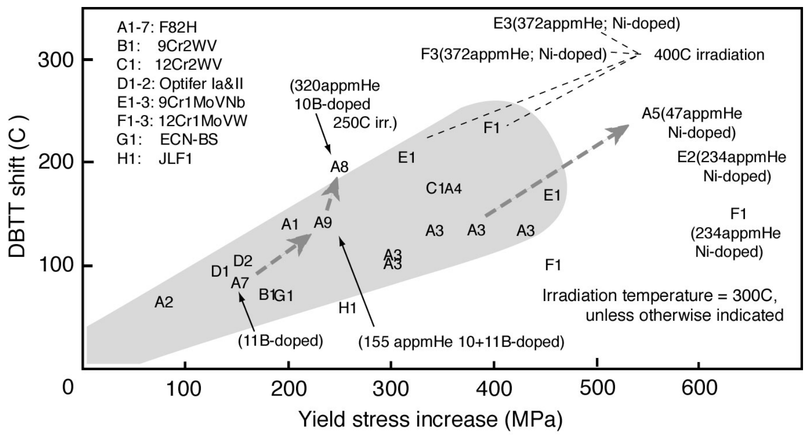


Fig. 4 ΔDBTT-$\Delta\sigma_{ys}$ relation for 7-12%Cr martensitic steels after irradiation. Some of the specimens were doped with B and Ni to introduce He atoms to evaluate the effect on DBTT.

5. Fatigue Properties

Fatigue properties of RAF/Ms before irradiation are almost similar to those of non-reduced activation commercial 9Cr-1MoVNb steels [6].
Because fatigue life follows residual ductility and the residual ductility after irradiation hardening is still rather high, it is expected that irradiation does not strongly reduce fatigue life. Therefore, one of the matters of interest is the irradiation induced mechanism change of fatigue damage. Post irradiation fatigue tests were performed for F82H at room temperature [17]. Irradiation was carried out in JMTR to 3.8dpa at 250C. Results are summarized in Fig. 5. Irradiation did not cause change in fatigue life, except for the results with smallest plastic

strain range of 0.04%. The number of cycles to failure decreased to one seventh that of the unirradiated specimen at the strain range. Based on the observations of fracture surface, it was suggested that the reduction might be attributed to channel deformation under cyclic stress.

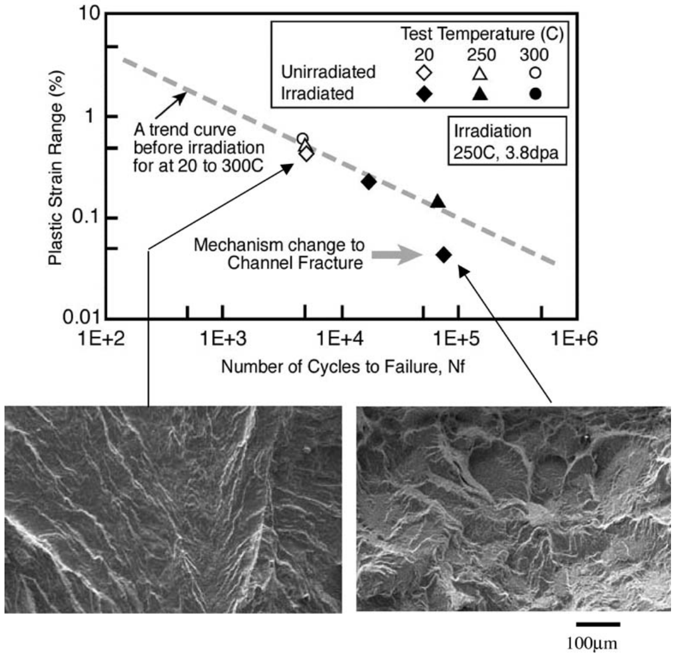


Fig. 5 Fatigue behavior after irradiation. Plastic strain range v.s. number of cycles to failure for irradiated F82H [17]. Irradiation reduced fatigue life by 1/7 at plastic strain range of about 0.04% (corresponded to 0.4% total strain range).

6. Compatibility

Corrosion resistance in high temperature water environments of RAF/Ms are reported to be almost similar to that of non-reduced activation martensitic alloys containing 9-12% Cr.
It is well known that the susceptibility for environmentally assisted cracking in high temperature water is relatively low for 9-12% Cr martensitic steels. On the other hand, it has been indicated that irradiation hardening often increases the susceptibility for IASCC of 316 stainless steel [18]. Slow strain rate tensile (SSRT) test in high temperature water environments was, therefore, carried out for irradiated F82H specimens to 2dpa in JMTR at 250C. Figure 6 shows the engineering stress-strain relation obtained during the SSRT tests. Almost no indication of environmentally assisted cracking was detected.

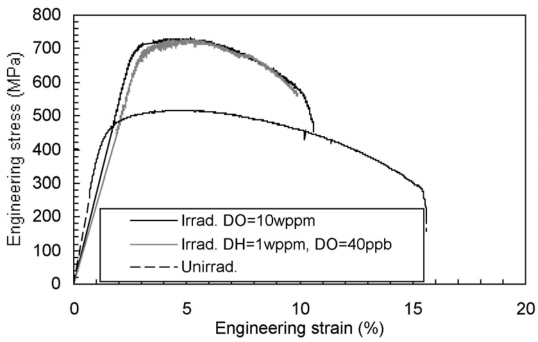


Fig. 6 Engineering stress-strain relations for SSRT tests performed in high-temperature water environments [18]. No obvious environmental effect was detected.

7. Materials Engineering and Design Methodology
7.1 Tensile Properties of Joints

Post irradiation tensile tests have been performed on TIG weld joint and HIP bonded specimens of F82H.
TIG joint specimens irradiated to 5dpa at 300C and 500C exhibited almost similar properties with base metal specimens, although hardening by irradiation for joint specimen at 300C was slightly smaller [14].

It has been reported that HIP process at 1040C for 5h with 150MPa hydrostatic pressure produced joints with strength comparable to base metal [19]. HIP joint tensile specimens were irradiated in JMTR to 1.5dpa at 250C and tested. Both strength and elongation of HIPed specimens were comparable to those of base metal specimens even after irradiation [20].

7.2 Effect of Temperature Change on Strength

Temperature of the blanket may change during start-up and flat-top of plasma operation. Temperature change during irradiation often causes to enhance irradiation hardening. As a preliminary investigation, tensile specimens of F82H were irradiated in JMTR to 1.5dpa during changing irradiation temperature cyclically between 250 and 350C. Dwell time at the each temperature was of about 50%. Total number of the temperature cycles was of ten. Extra-hardening was occurred by the temperature change during irradiation, as seen in Fig. 7. It should be pointed out that hardening often causes to reduce the margin to ductile fracture in strain.

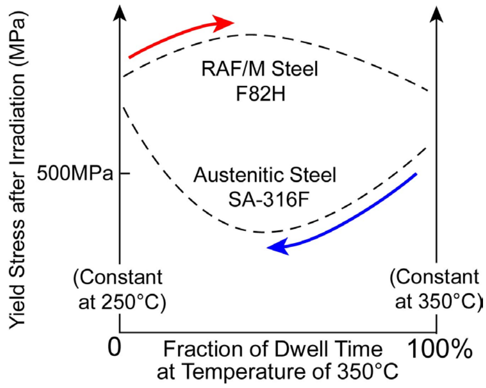


Fig. 7 Effect of temperature change on irradiation hardening of F82H. F82H exhibited extra-hardened by the temperature change between 250 and 350C, while an austenitic alloy (SA-316F) exhibited less hardening.

7.3 Margin to Ductile Fracture (Flow stress-strain relation after irradiation)

Ductile fracture condition is one of the key factors to estimate the margin to fracture of the component. Ductile fracture condition under the complex loading may be estimated from the constitutive equation of plastic flow stress-strain relation of the material. To evaluate the constitutive equation, an approximate true plastic flow stress-strain relation of irradiated F82H was obtained using a method to measure the neck development in the specimen during

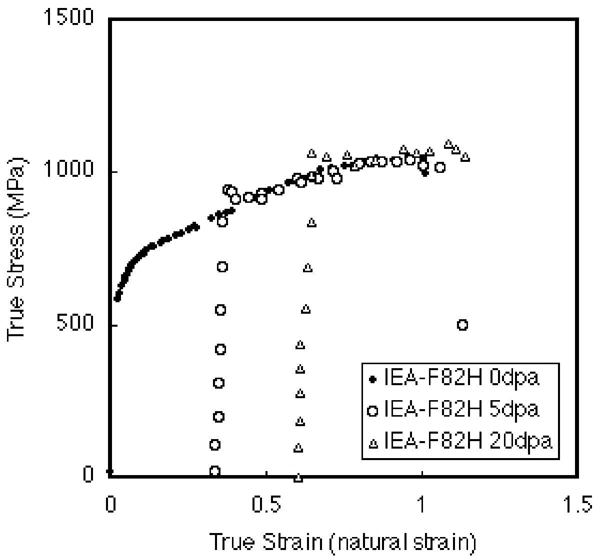


Fig. 8 Approximate true stress-true strain relation before and after irradiation [21]. The curves for irradiated specimens overlap well with those before irradiation, indicating irradiation does not affect the plastic behavior of F82H, other than hardening.

a tensile test [8,21]. A simple relation was obtained; the true stress-strain relation of the irradiated specimen was expressed well with an equation of $\sigma_{irr} = f(\varepsilon_0 + \varepsilon)$ in case that the constitutive equation for the unirradiated specimen is $\sigma_{un} = f(\varepsilon)$ (σ_{un} and σ_{irr} are flow stress before and after irradiation, ε is plastic strain and ε_0 is an equivalent strain for irradiation hardening) [8,21]. Indeed, the curve for the irradiated specimen overlaps well with that for the unirradiated specimen by shifting the curve along the strain axis, as seen in Fig. 8. Results suggest that hardened material by cold working may be used to estimate the deformation and fracture of the irradiation hardened component under monotonic loading, as far as the anisotropy introduced by cold working is negligible.

8. Oxide Dispersion Strengthened (ODS) RAF/M Steels

ODS RAF/M steels exhibit superior strength at elevated temperatures, as has been reported [4]. The steels have also better resistance to oxidation at elevated temperatures. Figure 9 shows the weight loss of different steels exposed to humid air at 750C. The oxidation resistance of 9Cr ODS RAF/M steel is much better than Martemsitic steels and is similar to 316 austenitic steel. The oxidation resistance of the 9Cr-ODS steel is attributed to homogeneous oxide film formed at the surface, which is considered to be due to suppression of oxygen diffusion by nano-sized yttria particles.

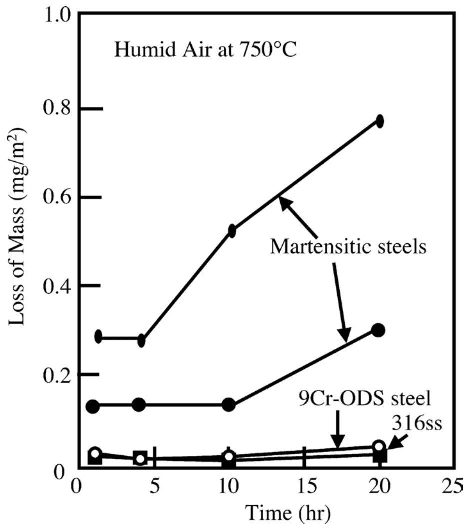


Fig. 9 Weight loss of 9Cr ODS RAF/M steel and Martensitic steels. ODS steel exhibited low corrosion rate in a high temperature water vapor environment.

9. Summary

(1) Progress has been made in the evaluation of the irradiation effects on tensile and fracture toughness properties to high damage levels. Results indicate that RAF/Ms developed in Japan have adequate properties for the application to the structural materials of DEMO blanket and ITER TBM.

(2) Preliminary investigations revealed the effects of irradiation on fatigue and environmentally assisted cracking in high temperature water were not significant within the damage level of ITER TBM condition.

(3) Some of the materials engineering subjects, such as irradiation effects on the tensile properties of joints, the effect of temperature change on irradiation hardening and the margin to ductile fracture after irradiation need to be investigated for the preparation of design code and licensing. Examinations on these subjects have been carried out.

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