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Recent results of the reduced activation ferritic/martensitic steel development

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Abstract

Significant progress has been achieved in the international research effort on reduced-activation steels. Extensive tensile, fracture toughness, fatigue, and creep properties in unirradiated and irradiated conditions have been performed and evaluated. Since it is not possible to include all work in this limited review, selected areas will be presented to indicate the scope and progress of recent international efforts. These include (1) results from mechanical properties studies that have been combined in databases to determine materials design limits for the preliminary design of an ITER blanket module. (2) Results indicate that the effect of transmutation-produced helium on fracture toughness is smaller than indicated previously. (3) Further efforts to reduce irradiation-induced degradation of fracture toughness. (4) The introduction of a post-irradiation constitutive equation for plastic deformation. (5) The production of ODS steels that have been used to improve high-temperature strength. (6) The method developed to improve fracture toughness of ODS steels.

1. Introduction

Since the last International Conference on Fusion Reactor Materials (ICFRM-10), considerable progress has been made in the continuing development of the reduced-activation ferritic/martensitic steels (RAF/Ms) for fusion applications. The objective of this paper is to review progress since ICFRM-10, but because of the extensive international effort involved and the limited space available for this presentation, it will only be possible to highlight a small amount of the work. The subject areas chosen for this review were to provide an indication of the scope of the work being performed and the progress that has been made. Evidence of this progress is further shown by the wide range subjects covered in the papers published in this proceedings.

2. International collaborations

International collaborations, such as those under the framework of the International Energy Agency

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(IEA) implementing agreement on fusion and the US DOE/JAERI HFIR collaborative experiment for fusion reactor materials development have been effective in accelerating the development of RAF/Ms [1–11]. The IEA collaboration started in 1992 as one of the activities of IEA collaboration for fusion materials development. Reference alloys of IEA-F82H (F82H), JLF1, and Eurofer 97 were supplied for the collaboration [7,8]. The F82H, a nominally Fe–8Cr–2W–0.14V–0.04Ta–0.1C (without N), was originally developed by the Japan Atomic Energy Research Institute and JFE (former NKK Corporation). Through a collaborative effort in the working group of the IEA collaboration, the chemical composition and heat treatment condition for a new IEA heat of F82H was arrived at, and two 5-ton heats were produced by JFE and distributed to collaborators in Japan, Europe, and the United States.

As a result of work in Japan, Europe, and the United States, mechanical and physical properties were obtained for the alloy, and a comprehensive database was prepared [9]. Based on the experience from the early collaboration on F82H and the European fast breeder research, Eurofer 97, a 9Cr RAF/M alloy (Fe–9Cr–1W0.2V–0.07Ta–0.03N–0.1C), was produced by the European Union. This steel contains lower tungsten and higher Ta than F82H [7]. Reduction of tungsten is expected to result in a higher tritium breeding ratio and a reduction of Laves phase formation. Laves phase can embrittle the steel [8]. A comprehensive database for Eurofer 97 is being prepared, based on the work by European laboratories.

Once the databases for F82H and Eurofer 97 were available, materials design limits were prepared for the preliminary design of ITER test blanket modules [10,11]. The design limits prepared from the databases include irradiation effects, although the damage levels for the data were rather limited. It was pointed out that irradiation mainly caused hardening; hence allowable stresses for the unirradiated condition might be used for designing at temperatures where materials exhibited irradiation hardening. Because the properties were rather similar to those of commercial modified 9Cr–1Mo martensitic steel, it was indicated that both alloys would be adequate for the components. It was also pointed out that the alloys were, therefore, almost ready to use for an ITER test blanket [11]; however additional evaluation of materials properties will be required for detailed analysis during licensing.

Mechanical properties of F82H, ORNL 9Cr2WTaV, JLF1 and Eurofer 97 with higher damage levels have been evaluated during experiments under USDOE/JAERI HFIR collaboration [1–9,12]. These results exhibit the good performance of the steels and also suggest that RAF/Ms are adequate for DEMO blanket application.

3. Materials properties

3.1. Tensile properties

Temperature dependences of the yield stress and ultimate tensile strength of Eurofer 97 are shown in Fig. 1 [11]. Tensile strength and ductility properties of Eurofer 97 are similar to those of F82H and other martensitic alloys containing 7–9% Cr. Fig. 1 also shows plots for irradiated specimens to 0.32 displacements per atom (dpa) at 350 °C. Irradiation caused an increase in strength at temperatures below 350 °C and the residual ductility (defined by reduction of area) maintained a rather high value, depending on the damage level. Although only limited damage levels are shown for the results in Fig. 1, it was pointed out that this level would be applicable for the ITER test module design [11].

Damage level dependence of yield stress for F82H irradiated in HFR and HFIR are shown in Fig. 2 [9,11]. Yield stress increased linearly with logarithm of the damage level to 10–20 dpa at temperatures ranging from 200 to 300 °C. Irradiation to higher dose levels in a fast breeder reactor on martensitic steels containing 9–12% Cr and by ion-beam irradiation on F82H suggested that irradiation hardening saturates with dose at a damage level of 10–20 dpa [13,14].

3.2. Fracture toughness properties

Irradiation hardening below 400 °C reduces fracture toughness, which includes a ductile-to-brittle transition temperature (DBTT) shift to higher temperatures [15–22]. Fig. 3 shows the damage level dependence of DBTT

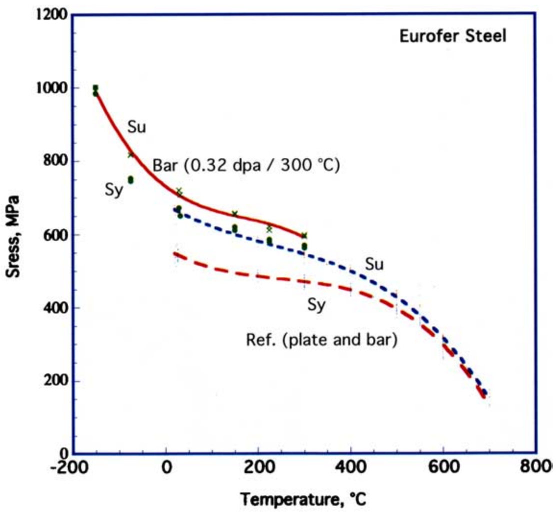

Fig. 1. Temperature dependence of yield stress and ultimate tensile strength for Eurofer 97. Irradiated results were obtained at SCK [11].

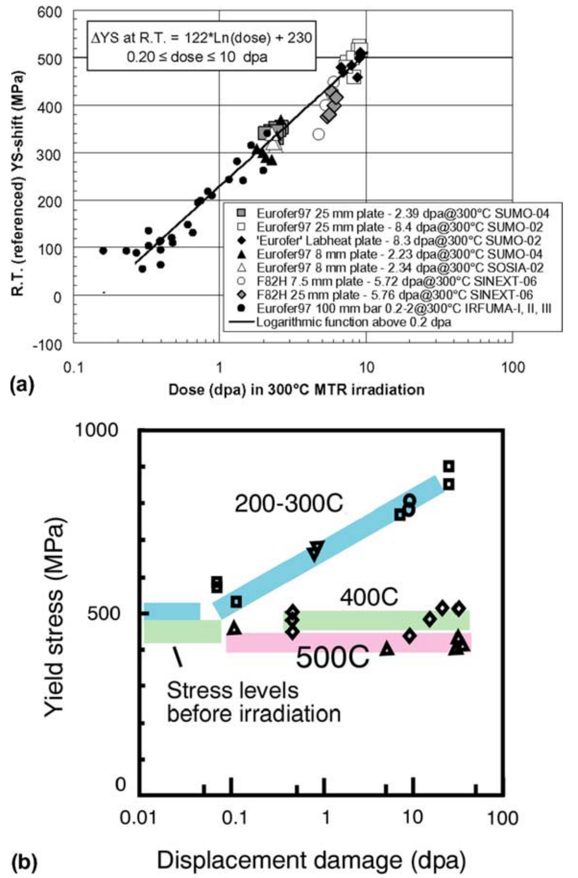

Fig. 2. Damage level dependence of F82H irradiated in (a) HFR [11] and (b) HFIR [17].

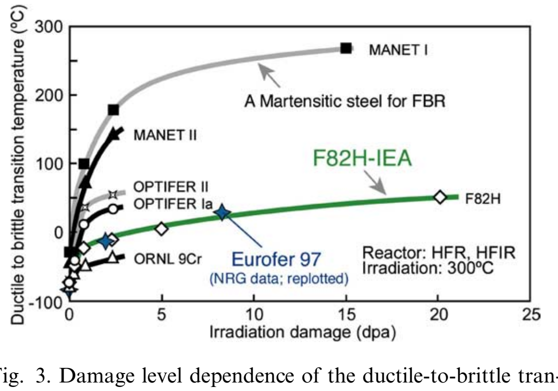

Fig. 3. Damage level dependence of the ductile-to-brittle transition temperature after irradiation at temperatures between 300 and 400 °C.

irradiated at temperatures between 300 and 400 °C. As seen in the figure, the DBTT shift tends to saturate with the damage level. When compared with shifts found in conventional (non-reduced-activation) steels, RAF/Ms exhibited smaller shifts, and for most properties, it appears that RAF/Ms exhibit similar or better irradiation performance than the conventional steels. Also, indications are that the DBTT shift caused by irradiation of RAF/Ms containing 9% Cr (e.g. Eurofer 97, JLF1 and ORNL 9CrWVTa) was smaller than that of F82H with 8% Cr (with less N but more W) [21].

It is desirable that for a water-cooled blanket application the DBTT does not exceed 100 °C. Although DBTT is readily affected by specimen size, F82H appears to satisfy this in the dose range up to several tens of dpa for Charpy tests (Fig. 3). However, results in Fig. 3 exhibit the effect of displacement damage only, and transmutation-produced helium atoms have been suggested to enhance the DBTT shift in a Charpy test [15–17]. The effect of helium on DBTT shift is discussed extensively in a later section.

Fig. 4 shows the temperature dependence of the fracture toughness of F82H before and after irradiation [18]. The DBTT after irradiation appears close to or higher than 100 °C (DBTT is sometimes determined to be the temperature at which fracture toughness satisfies 100 $MPam^{1/2}$). Results in Fig. 4 were obtained from compact-tension specimens with thicknesses of 5 and 10 mm. These results suggest a specimen size effect on DBTT. Therefore, care should be taken for the minimum dimension of the component used to examine the degradation of fracture toughness [9,23,24,51,52]. As far as the first wall of ITER is concerned, the thickness of the wall is similar or smaller than the ligament length of the specimens.

3.3. Fatigue properties

The relationship between total strain range and the number of cycles to failure for Eurofer 97 and F82H at temperatures of 250 and about 500 °C are shown in Fig. 5 [11,25]. Fatigue properties before irradiation are similar to those of conventional non-reduced activation

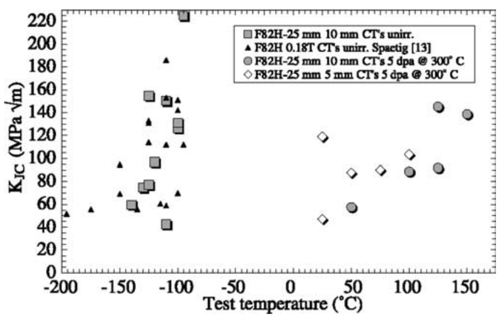

Fig. 4. Temperature dependence of fracture toughness of F82H before and after irradiation in HFR [18]. Compact-tension specimens with thickness of 5 and 10 mm were used; specimen size was larger than for results in Fig. 3.

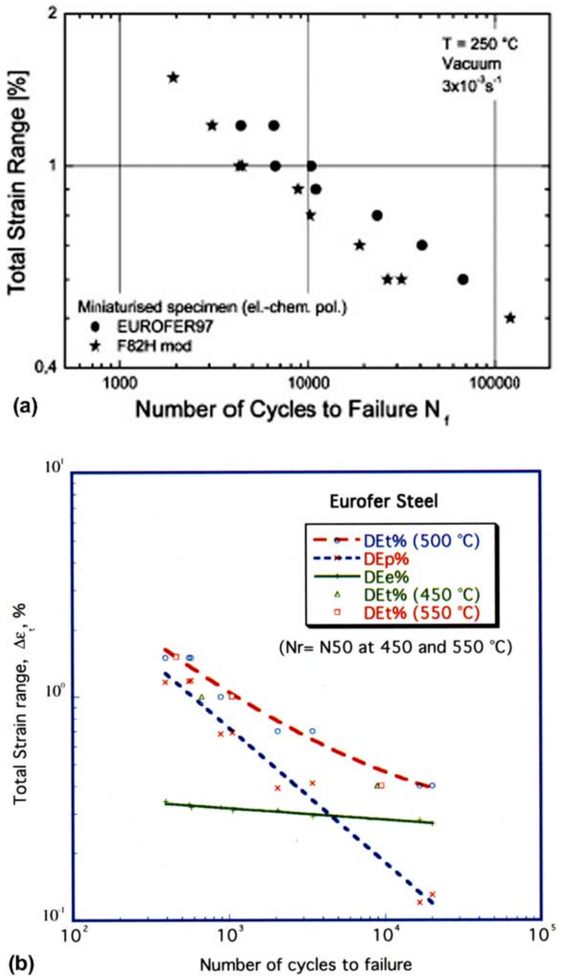

Fig. 5. Total strain range and the number of cycles to failure relation for Eurofer 97 and F82H [11]. Tests were performed at (a) room temperature and (b) ≈500 °C.

steels. Results showed that fatigue life for F82H was shorter than for Eurofer 97, which was due mainly to the larger content of non-metallic inclusions in the F82H fatigue specimen [25].

Knowledge of irradiation effects on fatigue behavior is increasing. It has been reported that irradiation to 3.8 dpa caused a change in the fracture mode during post-irradiation fatigue testing at room temperature with total strain range of 0.4% (0.04% of plastic strain range). The number of cycles to failure at room temperature decreased to one seventh that of the unirradiated material (Fig. 6) [26]. Based on observations of the fracture surface, it was suggested that that the reduction might be attributed to channel deformation under cyclic stress (see Fig. 6). A change of the fracture mode associated with a fatigue mechanism change has been reported for 316 stainless steel [27].

3.4. Creep properties

Fig. 7 compares the creep strength for Eurofer 97, F82H, and the Eurofer 97 oxide dispersion-strengthened (ODS) alloy (ODS steels are discussed in more detail in a later section) [11]. Creep strength of F82H was slightly higher than that of Eurofer 97. However, the ODS steel, strengthened with Y_2O_3 particles and using the Eurofer 97 as the matrix, had its strength increased quite effectively. Creep data under irradiation is limited, although it has been reported that irradiation strongly accelerated creep deformation of F82H at 590 °C [1]. Creep rate at the average temperature of the first wall of a water-cooled ITER blanket, however, is expected to be rather small.

3.5. Environmentally assisted cracking

Cracking under stress in a high-temperature pressurized water environment is one of the critical issues for austenitic steels. It has been also indicated that irradiation-induced segregation and hardening of 316 stainless steel caused an increase in the susceptibility for cracking [28]. Susceptibility for environmentally assisted cracking for martensitic steels is not believed to be an issue. However, there seems to be some possibility that severe irradiation hardening can assist cracking in RAF/Ms. Fig. 8 shows engineering stress–strain curves for irradiated and unirradiated F82H specimens in high-temperature water environments. No obvious effects of irradiation on the curves were observed [29]. Also, the fracture surfaces indicated that the specimens failed in a ductile manner.

3.6. Microstructure and microhardness

Void swelling caused by displacement damage to some tens of dpa is trivial for martensitic steels. Multi-ion beam irradiation with Fe, He (injection rate; 10–100 appm He/dpa) and H (injection rate; 20–200 appm He/dpa) has been performed for several martensitic steels and revealed that He and H atoms increased swelling at about 500 °C. It has been also reported that boron doping increased swelling of F82H by neutron irradiation. The swelling levels reported were, however, rather small, ≈1% or slightly higher [30].

Microstructural change at lower temperatures after irradiation by a spallation neutron source has been reported. Small loops, black dots, and small bubbles with sizes of 1 nm in diameter were observed to form after irradiation in PIREX and SINQ [31,32].

Large amounts of transmutation helium and hydrogen may cause the formation of a high number density of small cavities, which may cause additional hardening. Indications were that thousands of appm helium caused

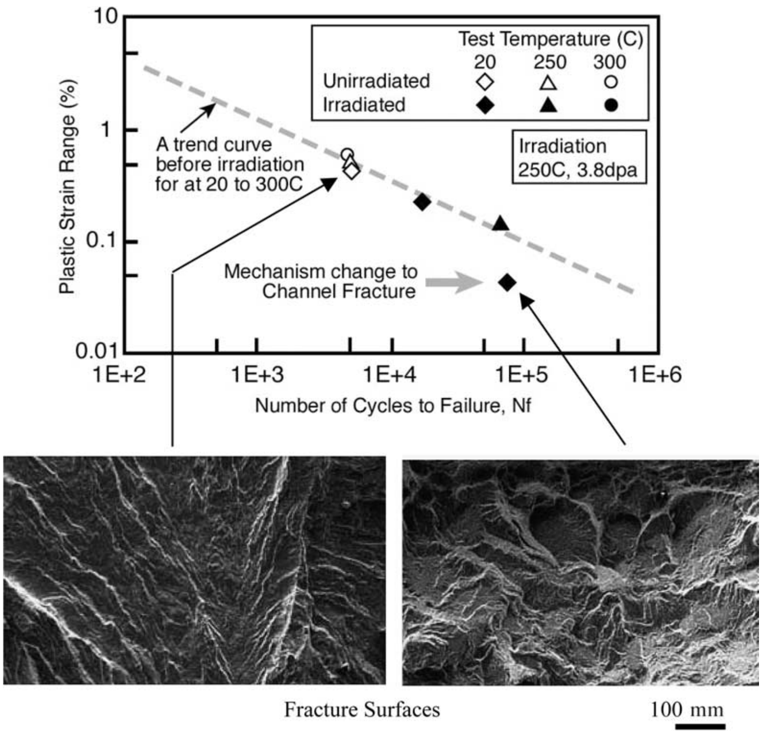

Fig. 6. Plastic strain range vs. number of cycles to failure for irradiated F82H [26]. Irradiation reduced the number of cycles to failure at room temperature by 1/7 at a plastic strain range of about 0.4%.

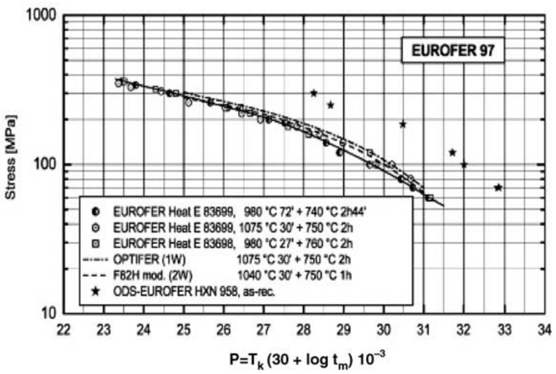

Fig. 7. Creep strength for Eurofer 97 and ODS Eurofer 97 with a curve for F82H [11].

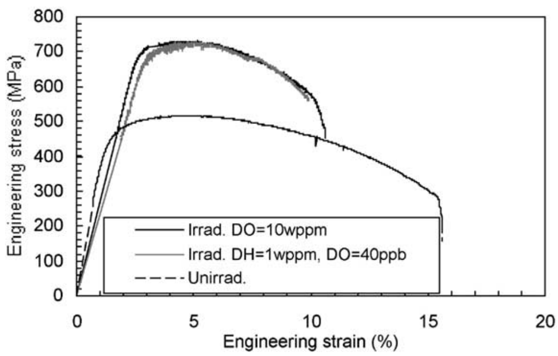

Fig. 8. Engineering stress–strain relations for slow strain-rate tensile (SSRT) tests performed in high-temperature water environments [29].

additional hardening during dual ion-beam irradiation [33].

Lower hardness after irradiation should lead to a lower DBTT shift. The effect of the tempering condition on the hardness after irradiation has been examined by ion irradiation. Results indicate that a higher tempering temperature (in the temperature range below the Ac1) and longer tempering time caused a reduction in hardness before and after ion irradiation. That is, the lower the hardness before irradiation, the lower the hardness after irradiation [34].

4. Effect of helium on the reduction of fracture toughness

Charpy tests on irradiated steels containing transmutation-produced helium atoms indicated that helium caused an increase in the DBTT shift [15–17]. For instance, from studies on irradiated B-doped and Ni-doped alloys, it was suggested that 100 appm He caused an additional DBTT shift by 150–200 °C. It was pointed out, however, that B and Ni doping may also introduce property changes in addition to the changes caused by helium [35].

There seems to be a systematic relation between DBTT shift and hardening by irradiation [5,22,36,53]. This suggests that the helium effect may be extracted by analyzing the relation from the results of B- and Ni-doped steels. Fig. 9 shows the relation obtained from the literature [15–17]. Because of the brittle fracture nature of the DBTT, the scatter in the plots is fairly large. The plots for B- and Ni-doped specimens are indicated in the figure. Also, arrows in the figure indicate the plots for results with and without doping.

For most of the results, there appears to be a relationship between the change in DBTT (ΔDBTT) and change in yield stress ($\Delta\sigma_{ys}$) such that ΔDBTT (in °C) = $A\Delta\sigma_{ys}$ (in MPa), where the proportional constant A is in the range 0.47 ± 0.19 (°C/MPa). This is mainly resulted from that DBTT is a function of flow stress, strain hardening capability, size of non-metallic phases, constraint at the crack tip, and the deformation rate. About half of the plots for the doped specimens are distributed in the region close to the upper bound in the shaded area in Fig. 9. Others remain in the region with an average value for *A* of the equation. Also, doping tended to be accompanied by additional hardening, as indicated by the arrows. The increased effect of helium seems to be about one third or smaller than has been indicated previously with He levels <400 appm. Simultaneously, suppression of hardening seems to be effective in reducing the DBTT shift. Although the results indicate that the additional hardening by some hundreds or thousands of appm helium may cause an additional DBTT shift [33,37,54], it would appear that more data are required to understand the helium effect.

5. Irradiation effect on the constitutive equation for plastic deformation

During tensile testing of irradiated specimens, the flow stress often drops just after plastic deformation begins. This stress drop occurs after a rather small strain (typically, <1% plastic strain). This effect has been attributed to dislocation channeling [38]. Dislocation channeling may lead low fracture toughness and reduction of fatigue life [26]. Because of the significance of this effect, a method for the computer simulation of dislocation channeling has been developed [39–42]. Also, a methodology was proposed to estimate a constitutive relation for plasticity from microstructure, taking the dislocation channeling into account [43].

Ductile fracture is often dominated by the strain hardening capability; localized necking to fracture occurs when $d\sigma/d\varepsilon = \sigma/2$ [44]. To evaluate strain hardening capability after irradiation over a wide strain range, an approximate true stress–true strain relation of irradiated F82H was obtained using a method to measure the neck developed in the specimen during a tensile test [45,55]. A simple relation was obtained; the true stress–true 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) [45]. Indeed, the curve for the irradiated specimen overlaps well with

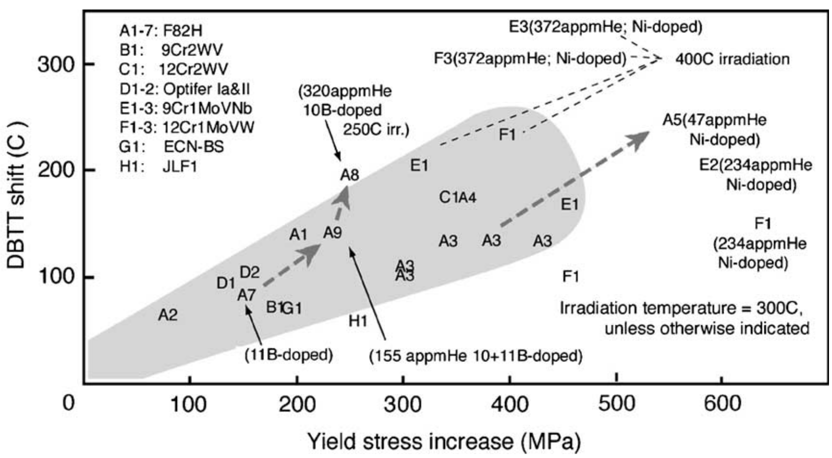

Fig. 9. ΔDBTT–σ_y relation for 7–9% Cr martensitic steels after irradiation. Some of the specimens were doped with boron and nickel to introduce helium in the materials to evaluate the effect of transmutation-produced helium on DBTT shift. σ_y values correspond to room temperature values converted from the values in the literature using the temperature dependence of the yield stress with and without irradiation [5,6].

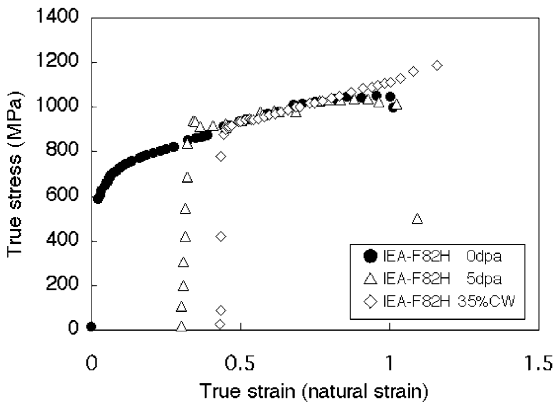

Fig. 10. Approximate true stress–true strain relation before and after irradiation [44]. The curves for irradiated specimens overlap well with those before irradiation, indicating irradiation does not affect the plastic behavior of the steel, other than hardening.

that for the unirradiated specimen by shifting the curve along the strain axis, as seen in Fig. 10. It is also important that the residual strain to fracture after irradiation was fairly large, and the margin for ductile fracture was not small.

6. Improvement of the high temperature strength

One of the issues for RAF/Ms for the application to a helium gas-cooled and liquid-metal-cooled blankets is the relatively low strength at high temperatures. Improvement of the strength by the addition of alloying elements, such as vanadium and nitrogen, has been carried out. Because limited elements are available to use and maintain the reduced-activation property, improvement of high-temperature strength by adjusting the chemical composition of the alloy is difficult. Oxide dispersion strengthening (ODS) by the addition of Y_2O_3 particles has been successfully applied to improve high-temperature strength of reduced-activation ferritic/martensitic steels [46–49]. Dispersion of a high number density of nano-size yttria particles is also effective to reduce radiation-induced microstructural change. A method by both hot isostatic pressing (HIPing) and hot extrusion to improve fracture toughness of ODS steels has been developed [49,56]. Dispersion of yttria is also reported to be effective in improving corrosion resistance at high temperatures [50].

7. Summary

(1) Mechanical properties, microstructures and physical properties of the reduced activation ferritic/martensitic (RAF/Ms) have been evaluated effectively by international collaborations. Databases for RAF/Ms have been prepared. The databases contain tensile, fracture toughness, fatigue, creep and other mechanical and physical properties.
(2) Databases are almost adequate for designing the ITER test module.
(3) One of the issues for RAF/Ms is the acceleration of the irradiation-induced decrease in fracture toughness by transmutation-produced helium. The effect, however, seems to be smaller than had been previously estimated.
(4) Progress has been made to establish a constitutive equation for plastic deformation after irradiation. An important observation is the knowledge that the margin for ductile fracture after irradiation is fairly large.
(5) Oxide dispersion strengthening is revealed to be effective in improving high-temperature strength of RAF/Ms. A technique to improve the fracture toughness of ODS steels has been also developed.

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