

**Material:** Ferritic Steel: F82H  
**Property:** Temperature (°C) versus 1T-adjusted  $K_{Jc}$  (MPa $\sqrt{\text{m}}$ )  
**Condition:** Irradiated  
**Data:** Experimental

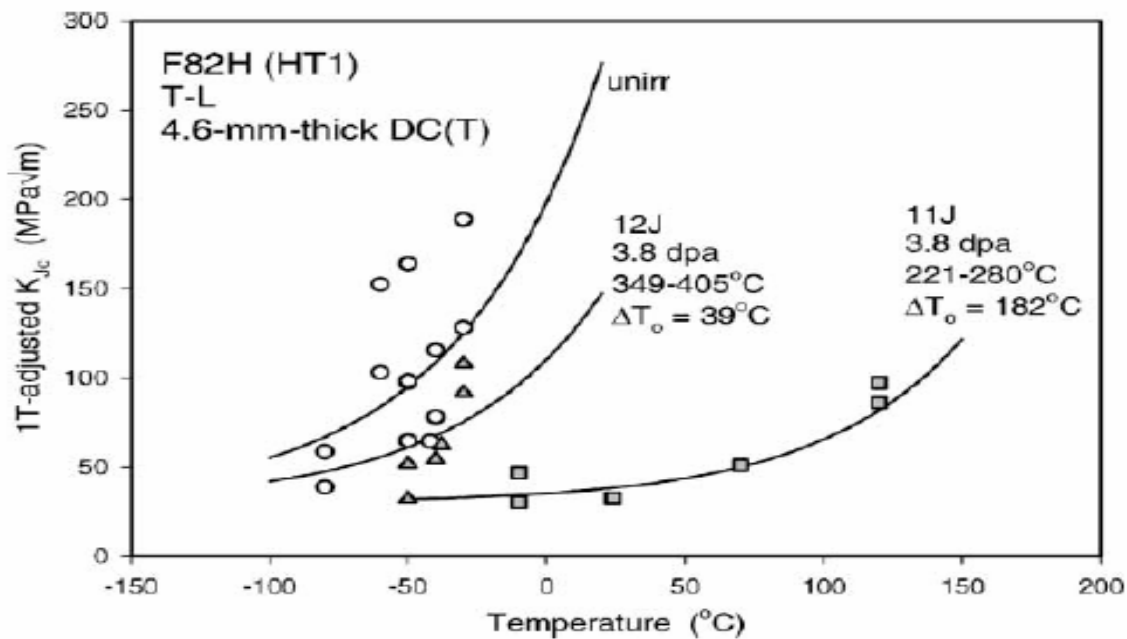


Fig. 1. Fracture toughness data for F82H as normalized and tempered (N&T) and after irradiation in HFIR at 221–280 and 349–405 °C to  $\approx 3.8$  dpa [11].

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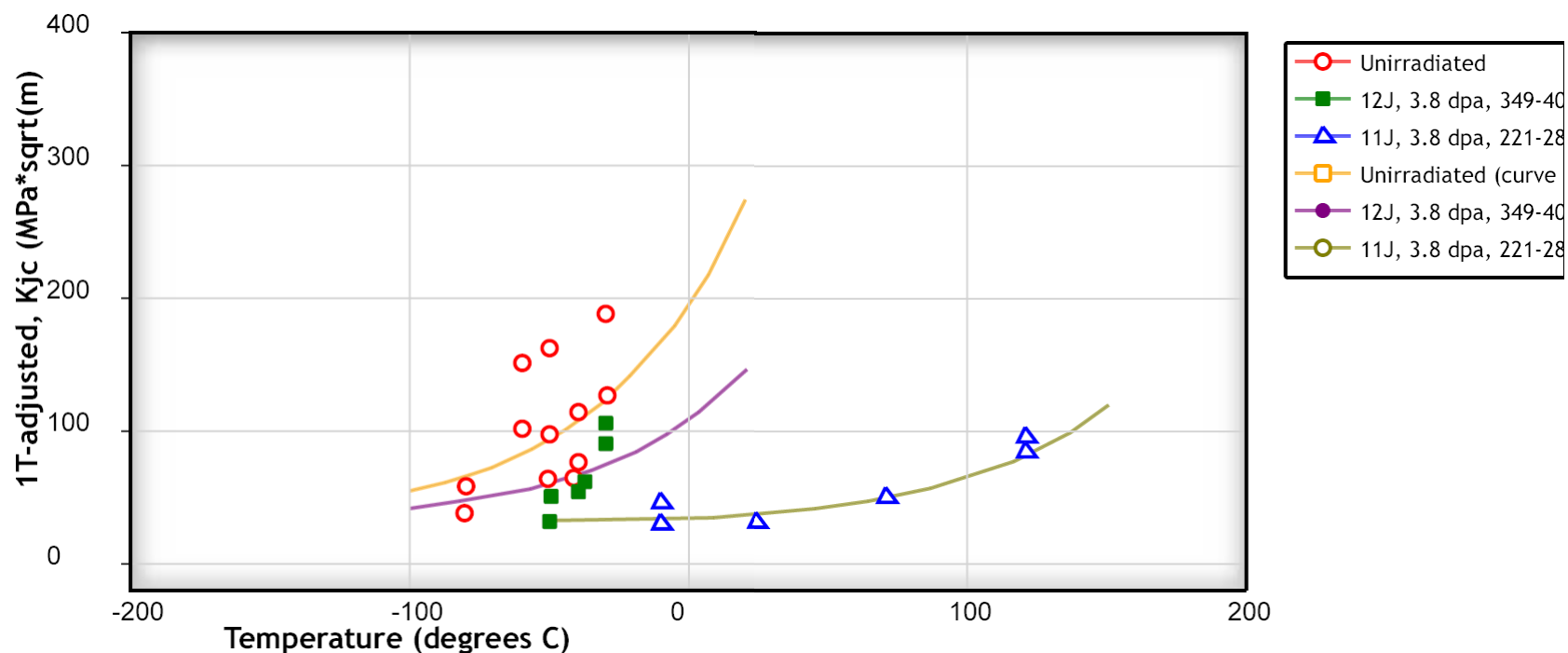
Ferritic/martensitic steels - overview of recent results

**Author of paper or graph:**

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**Caption:**

Fracture toughness data for F82H as normalized and tempered (N&T)  
 and after irradiation in HFIR at 221-280 and 349-405°C to approximately 3.8 dpa.  
 Note: All data for F82H (HT1) T-L 4.6 mm thick DC(T).



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#### Plot Format:

**Y-Scale:** ☒ linear ☐ log ☐ ln

**X-Scale:** ☒ linear ☐ log ☐ ln



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## Section 4. Ferritic/martensitic steels

## Ferritic/martensitic steels – overview of recent results

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B. van der Schaaf <sup>f</sup>, M. Victoria <sup>g</sup><sup>a</sup> Oak Ridge National Laboratory (ORNL), Metals and Ceramics Division, P.O. Box 2008, MS 6151, Oak Ridge, TN 37831, USA<sup>b</sup> PNNL, Richland, WA 99352, USA<sup>c</sup> JAERI, Tokai-Muro, Ibaraki-Ken 319-11, Japan<sup>d</sup> IAE Kyoto University, Gokasho, Uji, Kyoto 611-0011, Japan<sup>e</sup> UCSB, Santa Barbara, CA 93106, USA<sup>f</sup> NRG, Postbox 25, Petten, The Netherlands<sup>g</sup> PSI, CRPP-FTM, 5232 Villigen, Switzerland**Abstract**

Considerable research work has been conducted on the ferritic/martensitic steels since the last International Conference on Fusion Reactor Materials in 1999. Since only a limited amount of that work can be reviewed in this paper, four areas will be emphasized: (1) the international collaboration under the auspices of the International Energy Agency (IEA) to address potential problems with ferritic/martensitic steels and to prove their feasibility for fusion, (2) the major uncertainty that remains concerning the effect of transmutation helium on mechanical properties of the steels when irradiated in a fusion neutron environment, (3) development of new reduced-activation steels beyond the F82H and JLF-1 steels studied in the IEA collaboration, and (4) work directed at developing oxide dispersion-strengthened steels for operation above 650 °C.

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**1. Introduction**

Ferritic/martensitic steels were introduced into the fusion materials programs about 25 years ago [1], after research in fast reactor programs demonstrated their superior swelling resistance and excellent thermal properties compared to austenitic stainless steels. The first steels considered were conventional 9% and 12% Cr (all compositions are in wt%) Cr–Mo steels [2]. The development of reduced-activation steels began less than 20 years ago, and these steels are presently considered the primary structural material for a demonstration (DEMO) fusion plant and the first fusion power reactors.

Since the introduction of ferritic/martensitic steels into the fusion program, there have been questions concerning their applicability. An early concern and the reason the steels were not considered sooner involved the uncertainty associated with using a ferromagnetic material in the high magnetic fields in a fusion plant. The ferromagnetic material could cause a field perturbation on the plasma and, in turn, the high magnetic fields of the reactor could cause magnetostatic forces on the ferromagnetic structure. Preliminary calculations indicated that these problems could be handled in the reactor design [3,4]. Recent more detailed calculations along with continuing experimental work is verifying the earlier conclusions [5–8].

A second reason for questioning the use of the steels involved the feasibility of using them in the harsh neutron radiation environment of a fusion plant. Neutron irradiation at  $\lesssim 425$  °C hardens the steels, which affects the mechanical properties and fracture behavior. Of special concern is embrittlement and the effect of helium

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on embrittlement, as manifested in Charpy and fracture mechanics testing by an increase in the ductile-brittle transition temperature (DBTT) and a decrease in the upper-shelf energy (USE). That concern has been the subject of much work, and it is a continuing concern that will be discussed here.

The original objective of this paper was to give an overview of the progress made in the research on the ferritic/martensitic steels for fusion since ICFRM-9 two years ago. However, as the list of papers at ICFRM-10 shows, considerable progress has been made, and it is impossible to discuss all that work in a single paper. Instead, the following topics have been chosen for this discussion: (1) progress made in the international collaboration on the large heats of F82H and JLF-1, (2) helium-effects investigations, (3) the production of the large heat of EUROFER 97 and preliminary experimental results and (4) work on developing new steels to operate above the 550–600 °C limit of the reduced-activation steels.

## 2. International collaboration

At an International Energy Agency (IEA) sponsored Workshop for Ferritic/Martensitic Steels for Fusion in Tokyo in 1992, a proposal was made for an international collaboration on determining the feasibility of using ferritic steels for fusion. The Japanese delegation at the meeting proposed to make available large heats of ferritic/martensitic steels that could be used in a collaboration between Japan, Europe, and the United States. In subsequent IEA meetings, a modified F82H composition was determined, and in 1993 a 5-ton heat of this IEA F82H (Fe–7.5Cr–2W–0.2V–0.4Ta–0.1C) steel was ordered by the Japan Atomic Energy Research Institute (JAERI) and produced and processed into 7.5- and 15-mm-thick plates by NKK Corporation. This 5-ton heat has been referred to as F82H-Mod and F82H-IEA to distinguish it from F82H previously produced by JAERI and NKK, which has sometimes been called F82H-Standard. In this paper, the F82H-IEA steel will be discussed, but since it is basically the only heat discussed, it will be referred to simply as F82H. In 1995, a second 5-ton heat was produced by NKK and processed into 15- and 25-mm-thick plates that were used primarily to make tungsten inert gas (TIG) and electron beam welds. In addition to the F82H, a 1-ton heat of JLF-1 (Fe–9Cr–2W–0.25V–0.1C) steel was ordered by the Japanese Universities Fusion Materials Program and produced and fabricated into plates by Kobe Steel. Because of the lesser amount of material available, most of the work on that heat was carried out by Japan universities investigators.

The testing phase for the large heats of F82H and JLF-1 – especially for the unirradiated physical and

mechanical properties – is nearing completion. Physical properties data obtained were: density, specific heat, thermal expansion, thermal conductivity, Young's modulus, modulus of rigidity, Poisson's ratio, and magnetic hysteresis. Mechanical properties obtained were: tensile, creep, Charpy impact, fracture toughness, isothermal fatigue, thermal fatigue, and low-cycle fatigue. A computerized database has been developed that is available to the international community. A summary of the work will be published [9].

In addition to determining the baseline data, the F82H has been included in over twenty neutron irradiation experiments conducted in the High Flux Isotope Reactor (HFIR) in the US, in the Japan Research Reactor (JRR-4) and the Japan Materials Test Reactor (JMTR) in Japan, and the High Flux Reactor (HFR) in The Netherlands [10]. The results for the large heat of F82H were in general agreement with results for other experimental heats of 7–9Cr–WV-type reduced-activation steels that show improved properties over conventional Cr–Mo steels.

Studies on the F82H-IEA are continuing to determine the effect of irradiation on a range of properties for base metal and weldments. As discussed above, irradiation embrittlement is observed as an increase in DBTT in a Charpy test. Charpy data cannot be used for design purposes. Instead, fracture toughness data are required, and such data for irradiated F82H have been obtained recently (Fig. 1) [11]. In this work, six disk-compact tension [DC(T)] specimens were irradiated in HFIR to  $\approx 3.8$  dpa at two temperatures. Three of the specimens in the low-temperature capsule were at an average of 261 °C and another three at 240 °C. In the high-temperature capsule, all six specimens were irradiated at an average temperature of 377 °C. Fracture toughness transition temperatures were evaluated for irradiated F82H and

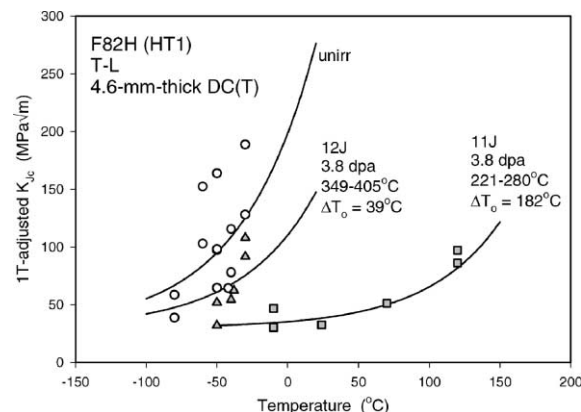


Fig. 1. Fracture toughness data for F82H as normalized and tempered (N&T) and after irradiation in HFIR at 221–280 and 349–405 °C to  $\approx 3.8$  dpa [11].

compared to unirradiated values (Fig. 1). The master curve concept was used to evaluate the shift of the fracture toughness transition temperature [11]. Specimens irradiated at the higher temperature exhibited a relatively modest shift of about 40 °C. However, the shift of fracture toughness transition temperature of specimens irradiated below 300 °C was much larger – about 180 °C. This shift is in general agreement with a DBTT shift observed for F82H Charpy specimens irradiated in HFIR at 300 °C [12].

### 3. Helium effects

The effect of the transmutation helium generated in the structural material of a fusion reactor first wall on the mechanical properties – especially the fracture properties – has been a constant source of uncertainty that makes the need for a 14 MeV neutron source urgent for materials studies. In the absence of such a source, simulation experiments using ion implantation or  $^{54}\text{Fe}$ -, Ni-, or B-doping are the primary techniques used to study helium effects on mechanical properties during neutron irradiation. Studies are also possible with irradiation in a spallation neutron source or a proton irradiation facility, although until recently, most of the studies in these facilities have concentrated on swelling and radiation damage. All these techniques, with their inherent problems and uncertainties, are now being used to further our understanding of a possible helium effect on mechanical properties.

Studies using ion implantation [13], nickel-doping [14], and boron doping [15–17] have all produced results that were interpreted to indicate that helium increases the shift in DBTT over that due to displacement damage alone. Results with these techniques have also indicated an increased amount of swelling, and recent results [18,19] have reiterated such a relationship when F82H with  $\approx 30$  and  $\approx 60$  ppm natural boron and  $\approx 65$  ppm  $^{10}\text{B}$  was irradiated in HFIR at 400 °C up to 52 dpa to produce  $\approx 30$ ,  $\approx 60$  and  $\approx 330$  appm He, respectively (Fig. 2). Helium is produced according to the reaction:  $^{10}\text{B}(n, \alpha) ^7\text{Li}$ ; natural boron contains  $\approx 20\%$   $^{10}\text{B}$ .

Cavity size, number density, and swelling after 52 dpa for the three steels were respectively 12.7 nm,  $6.1 \times 10^{20} \text{ m}^{-3}$ , and 0.52%; 10.6 nm,  $2.4 \times 10^{21} \text{ m}^{-3}$ , and 1.2%; and 7.6 nm,  $6.1 \times 10^{21} \text{ m}^{-3}$ , and 1.1%. The swelling of 1.2% and 1.1% after only 52 dpa for the steels with 60 and 330 appm He, respectively, is much larger than generally expected for these steels. The higher density of smaller cavities for the steel with 330 appm He than the steel with only 60 appm He evidently suppressed the swelling by acting as neutral sinks compared to the lower density of larger cavities in the steel with 60 appm He. Most previous high-dose data that show low swelling come from fast reactor irradiation (little helium

generated), and as seen in Fig. 2(d), the swelling of F82H irradiated in the fast flux test facility (FFTF) to 67 dpa ( $< 5$  appm He) was about an order of magnitude less (0.12%) [20]. Such large swelling as observed in this experiment again emphasizes the need for a 14 MeV source for materials development.

All the simulation techniques have problems. Boron, for example, is a reactive element that can be associated with precipitates and prior austenite grain boundaries; also, lithium from the transmutation of  $^{10}\text{B}$  could cause problems. Furthermore, during fission reactor irradiation, all the boron is quickly transformed into helium, which differs from what happens in a fusion reactor, where helium forms simultaneously and more gradually with the displacement damage.

When nickel-doped steels are irradiated in a mixed-spectrum reactor, displacement damage and helium form simultaneously, although this technique also has its problems [21,22]. In recent work, a 9Cr–2W reduced-activation steel with and without 1% Ni was irradiated to 2.2 and 3.8 dpa at 270 and 348 °C, respectively, in the advanced test reactor (ATR). The nickel-containing steel hardened about 20% more than the steel without nickel at 270 °C, but strengths were similar after irradiation at 348 °C [23]. Likewise, there was a larger shift in DBTT for the nickel-containing steel than the one without nickel when irradiated at 270 °C, but not after irradiation at 348 °C. TEM analysis indicated that nickel refined the size of the defect clusters, which were more numerous in the nickel-containing steel [23].

These results indicate that the nickel-doping simulation technique should be used with caution below about 300 °C. The results using nickel doping that most strongly indicated that helium caused an increase in the DBTT above that caused by displacement damage alone were high-dose tests at 400 °C [14]. Tensile tests at this temperature gave no indication of hardening due to helium (or nickel) [14]. TEM studies of nickel-doped steel irradiated in HFIR and FFTF showed that a high density of  $\text{M}_6\text{C}$  formed in the nickel-doped steel but not in the undoped steel [24]. Since the shift in DBTT in HFIR, where helium forms, was larger than in FFTF, where little helium forms, the results were taken to mean that helium caused the shift [14].

One simulation technique that bypasses problems such as those associated with B- and Ni-doping is the replacement of the natural iron in the steel with  $^{54}\text{Fe}$  [25–27]. Transmutation of  $^{54}\text{Fe}$  when irradiated in HFIR produces helium and hydrogen in the steel. TEM disks with 96% of the natural iron in F82H replaced with  $^{54}\text{Fe}$  were irradiated in HFIR to 34 dpa to produce 65 appm He and 400 appm H. Mechanical properties were obtained from the 3-mm disks with a shear punch technique that can be used to study hardening. There was some indication that hydrogen might affect the strength [25], but no effect of helium could be detected [25–27].

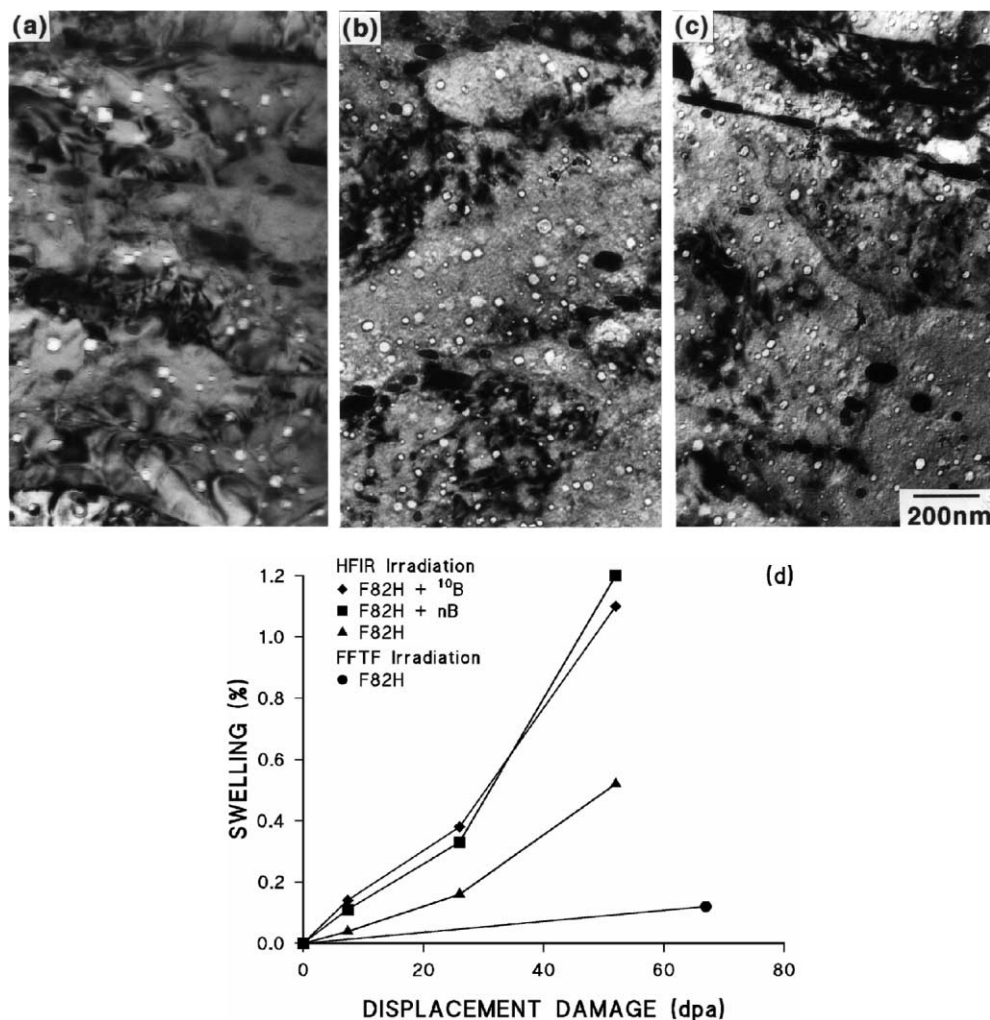


Fig. 2. Cavities in normalized-and-tempered F82H with different boron levels irradiated to 52 dpa in HFIR at 400 °C to produce (a) 30, (b) 60, and (c) 330 appm He, and (d) swelling as a function of displacement damage for the F82H steel with different helium concentrations irradiated in HFIR compared to that irradiated in FFTF [18].

This shear-punch test procedure is a good example of the sophistication that has been achieved in recent years in the fusion materials programs on the miniaturization of mechanical properties testing. The punch test produces a 1-mm disk from the center of a conventional 3-mm TEM disk that can then be thinned for TEM examination. In addition, the 3-mm OD ring can be used to analyze for helium and hydrogen concentrations [25].

The use of  $^{54}\text{Fe}$  is superior to the other simulation techniques for single-variable experiments to study helium. However, the isotope is expensive ( $\approx \$20\,000\text{ g}^{-1}$ ), and thus, the cost of making even miniature tensile, Charpy, and other specimens may be prohibitive. Another problem is that the He/dpa production ratio is about 2.3 for a fission neutron spectrum, compared to a value of around 10 for a fusion neutron spectrum. This

ratio could be improved by using  $^{55}\text{Fe}$ , but this isotope is even more expensive than the  $^{54}\text{Fe}$  [28].

A small-punch test technique was also used to test 9Cr–2W steel implanted with  $\alpha$ -particles up to 580 appm He and 0.226 dpa at 80–150 °C [29]. The implantation caused an increase in the DBTT estimated at 98 °C for a standard Charpy V-notch specimen through the conversion of small punch test results by an empirical relationship. The increase in DBTT was interpreted as a displacement-damage effect, suggesting that there was no significant effect of helium for these test conditions.

Another method to simultaneously produce helium and displacement damage is irradiation with 600–750 MeV protons in a facility such as PIREX at PSI (Paul Scherrer Institute) [30]. To investigate the location of helium during low-temperature irradiation, pure iron

and F82H were irradiated with 600 MeV protons at 50–350 °C in PIREX and with neutrons in the DR-3 experimental reactor. Copper was also irradiated to compare bcc and fcc structures. Positron annihilation spectroscopy was used to study the evolution of vacancy clusters.

The displacement damage in neutron-irradiated iron and F82H produced a cavity structure over the entire temperature range, and there was a difference between iron and copper [30]. Cavities nucleated in copper only above  $\approx 200$  °C, but they nucleated in the iron and steel at 50 °C. When helium was generated in the proton irradiation for doses similar to the neutron irradiation, a similar cavity structure formed. From these observations, the fact that helium does not produce hardening, an observation made in numerous experiments, can be explained. Namely, helium is distributed in the pre-existing cavities (produced by displacement damage). Cavity structures similar to those observed in the iron were also observed in F82H, indicating a similar explanation applies to the more complicated alloy. This observation of a lack of hardening caused by helium in the steels complicates any explanation of the increase in DBTT of these steels that has been attributed to helium. The only time helium should have an effect on strength in this case would be for high helium concentrations where high-pressure bubbles could form [30]. Such hardening by helium has been demonstrated in MANET [31] and F82H [13] implanted with up to 500 appm He.

Results coherent with those from PIREX were observed when F82H was irradiated at 90–390 °C to 2.8–12.5 dpa with protons in the Swiss Spallation Neutron Source (SINQ) [32]. Again, helium was produced simultaneously with displacement damage, with helium ranging from 180 to 818 appm. Small bubbles near the limit of resolution (average size estimated at 0.95 nm) were observed by TEM after 5.8 dpa (210 appm He) at 217 °C. Bubble size increased with irradiation temperature, but the number density remained relatively constant at  $\approx 4 \times 10^{23} \text{ m}^{-3}$ . After 12.5 dpa (820 appm He) at 390 °C, the size was  $\approx 1.6$  nm, corresponding to swelling of  $\approx 0.06\%$ . Although the He:dpa production rate is much higher than in a fusion reactor, helium can nucleate bubbles even at these low temperatures. It is at these temperatures where observations of a helium effect on the impact properties have been observed in the simulation experiments.

#### 4. Steel development

Reduced-activation steel development began with small experimental heats to determine compositions with mechanical properties as good or better than the Cr–Mo steels they were to replace. Once that was achieved, the large heats of F82H-IEA and JLF-1 were

used to establish the feasibility using of the steels for fusion. The next step was to use what was learned to produce an advanced steel, and the European fusion materials program has taken that step and produced a 3.5-ton heat of a steel designated EUROFER 97 [33]. The heat was ordered in 1997, and bar, plate, tubing, and wire were delivered in 1999.

Nominal target chemistry for EUROFER was Fe–9.0Cr–1.1W–0.2V–0.07Ta–0.03N–0.11C, which differs from the F82H-IEA that had a target of Fe–8.0Cr–2W–0.2V–0.04Ta–0.1C [34]. Differences from the target composition in the F82H were that it contained less Cr (7.5%), Ta (0.023%), and V (0.14%) than the target composition; the EUROFER contained more tantalum (0.14%) than the target composition [35]. The major differences in the steels are the Cr, W, and Ta compositions. Lower tungsten was used in EUROFER for neutronic reasons, but it will also reduce the possibility of Laves phase formation relative to the steels with higher tungsten.

Despite the differences in composition of EUROFER 97 and F82H, the two steels had similar tensile (Fig. 3), impact (Fig. 4), and creep properties [36,37]. The data on the impact properties indicate that the EUROFER has a lower DBTT and a similar to slightly lower USE

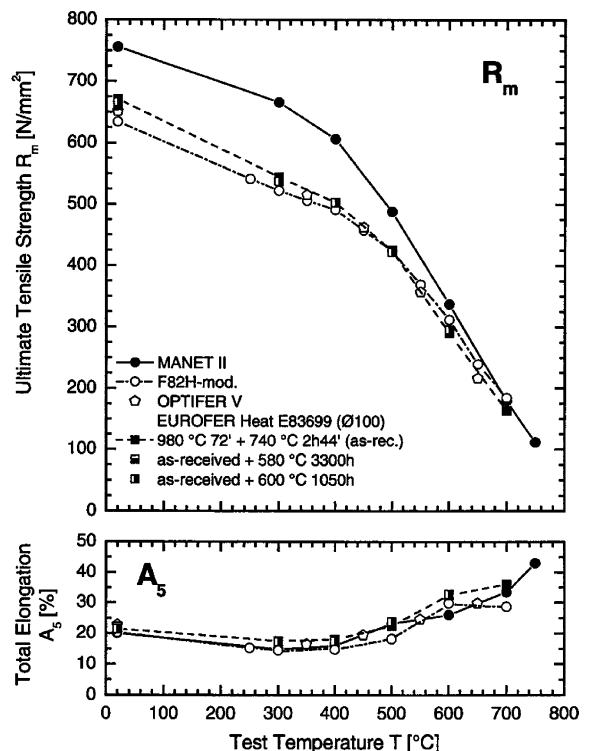


Fig. 3. Ultimate tensile strength and total elongation of EUROFER 97 compared to F82H, OPTIFER, and MANET II [35].

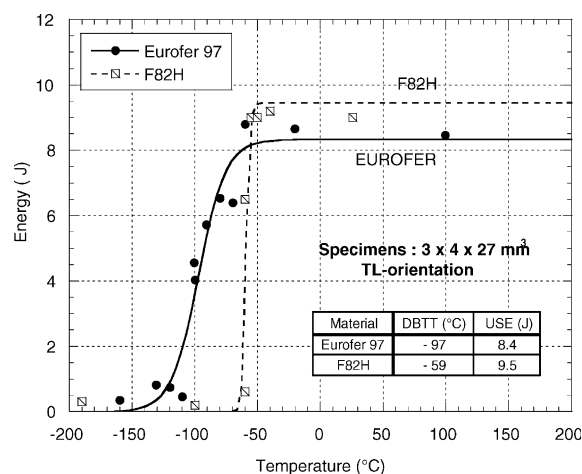


Fig. 4. Impact behavior of EUROFER 97 compared to F82H [36].

[36,37]. Heat treatment (austenitizing and tempering temperatures and times) affected the impact and creep properties [36].

## 5. Development of advanced ferritic/martensitic steels

An interesting observation on the tensile behavior of EUROFER is the higher strength of the MANET II (Fig. 3), a nominal Fe–10Cr–0.6Mo–0.7Ni–0.2V–0.15Nb–0.1C steel. One of the objectives in developing the reduced-activation steels was to produce steels with properties similar to the conventional Cr–Mo steels they were to replace. If the results in Fig. 3 are representative, this was not achieved for the F82H and EUROFER 97 with respect to MANET II, indicating that there is still room for improvement. This conclusion is also supported by the improvement obtained in recent years in the elevated-temperature strength of conventional Cr–Mo steels [38]. Using selective alloying, operating temperatures for the Cr–Mo steels used for power-generation applications have been pushed to 600–625 °C, compared to the 550–600 °C generally attributed to steels such as modified 9Cr–1Mo and the reduced-activation ferritic/martensitic steels. This indicates that improvement of reduced-activation steels by conventional alloying techniques may be possible.

To examine the possibility of improving the properties of reduced-activation steels, work in Japan examined the effect of tungsten on 9Cr–xWVTa steels by examining steels with nominally 2.0%, 2.5%, 3.0%, and 3.5% W (the steel with 2% W is JLF-1) [39]. Tungsten caused an increase in strength and a slight decrease in total elongation when increased from 2.0% to 2.5%, with little change for a further increase to 3.5%. Increasing

the tungsten from 2% to 3.5% caused a large increase in the DBTT and a reduction of the USE, although in another experiment, a steel with  $\approx 2.4\%$  W had a significantly lower DBTT than one with 2% W [40]. The creep-rupture properties were improved by increasing the tungsten content, although this is contrary to the results on the EUROFER 97 (1% W) and F82H (2% W), which had similar creep-rupture properties [36,37].

Precipitates were analyzed in the 2.0% and 3.0% W steels after normalizing and tempering and after a short-time creep-rupture test at 700 °C and a long-time test at 650 °C [39]. The main difference was that Laves phase precipitated in the two creep tests of the 3.0% W steel, but Laves only precipitated in the long-time test for the 2.0% W steel. The 3.0% W steel had slightly more tungsten in solution after the long-time rupture. Since the long-time test for the 3.0% W steel was only 1650 h and tungsten in solution is thought to enhance creep strength, it may be that for longer times more tungsten will be lost from solution, and the enhanced creep-rupture strength will be lost for tests to  $10^5$  h and beyond. The results indicate the complicated nature of the effect of composition and of using composition changes to improve properties.

## 6. Oxide dispersion-strengthened steels

One attractive route to materials capable of higher operating temperatures and that maintain the advantages of the ferritic/martensitic steels would be the development of oxide dispersion-strengthened (ODS) steels. That option is being pursued in Europe, Japan, and the United States. Development of ODS steels for fast reactor cladding began in Belgium in the late 1960s, and work for that application has continued [41,42]. The main problem that kept them from being used was the anisotropy in properties that results from the way the steels are fabricated (mechanical alloying of powders, compaction, and extrusion). The problem with anisotropy still exists in more recent steels being developed [41], although there have been advances. Most of the early studies on the ODS steels concentrated on the creep and tensile behavior [41–43].

Recently, fracture properties of a commercial MA 957 rod were determined, and the anisotropy was observed in terms of a 10:1 grain aspect ratio [44]. Three orientations relative to the rod axis (defined in Fig. 5) were tested. Tensile tests between –150 and 150 °C indicated that the yield stress of the C–L and C–R specimens were higher than the L–R specimens up to about room temperature, after which the C–R and L–R specimens had similar strengths, which were greater than those of the C–L specimens. The ultimate tensile stresses of all three orientations were similar. Uniform and



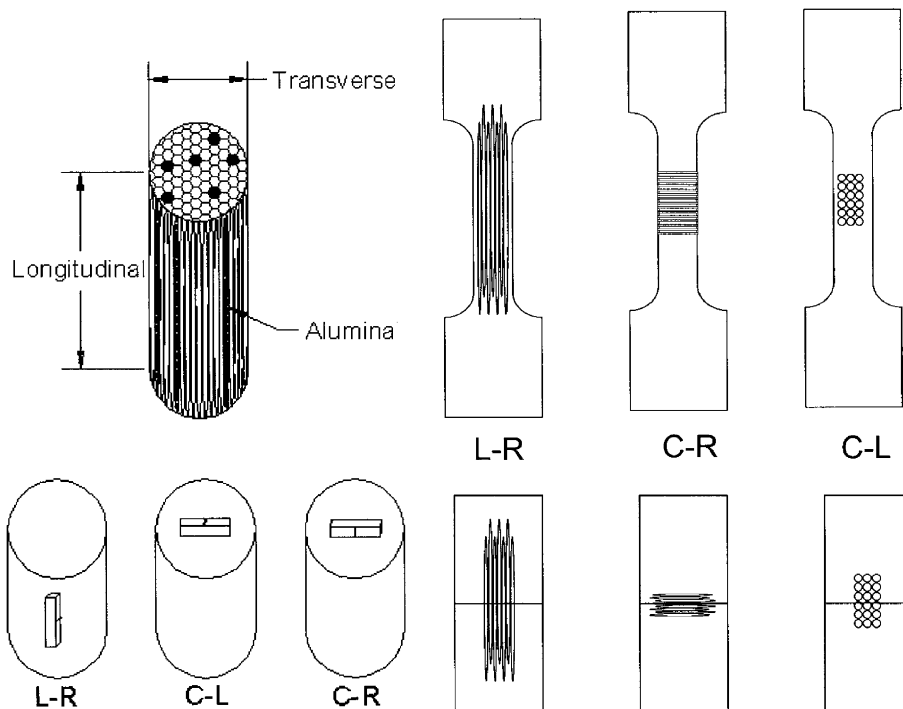


Fig. 5. Schematic diagram showing orientation of test specimens from an MA957 rod [44].

fracture plastic strains of the L-R orientation were much higher than the other two orientations.

Toughness measurements indicated the L-R orientation was superior to the other two orientations, which were similar (Fig. 6). Fractography indicated that the reason for this behavior was alumina ( $\text{Al}_2\text{O}_3$ ) stringers parallel to the long grains produced during processing. Toughness was highest in the direction orthogonal to the short grain/stringer axis and lowest parallel to long grain/stringer axis. It was concluded that  $\text{Al}_2\text{O}_3$  particles triggered cleavage cracks that induced the low toughness in the C-R and C-L orientations. The obvious solution is the elimination of the alumina stringers and a reduction of the grain aspect ratio. A development program to achieve those goals is planned [44].

ODS steel development programs are also in progress in Europe [45–48] and Japan [49]. To achieve isotropic microstructures and properties, two procedures are employed: (1) produce ferritic ( $\geq 12\%$  Cr) steels and by mechanical processing and heat treatment develop equiaxed microstructures, and (2) produce martensitic (7–10% Cr) steels that form equiaxed structures during the austenite-to-martensite transformation. The use of hot isostatic pressing (HIP) to fabricate the ODS steels is also being pursued.

In Europe, much of the development effort is on a EUROFER ODS composition that will be produced as a martensitic steel because of its composition. EURO-

FER powder has been manufactured, mechanically alloyed with  $\text{Y}_2\text{O}_3$  powder, and consolidated and formed by HIP or extrusion to produce the ODS material [45–48]. Much of the early effort has been on producing sound material. Early tensile properties indicate that EUROFER ODS has a much higher 0.2% yield stress and excellent uniform elongation relative to conventionally produced EUROFER and F82H (Fig. 7) [48]. Preliminary room-temperature impact results are also encouraging [44].

The Japanese ODS program involves work at JNC (Japan Nuclear Cycle Development Institute), Kyoto University, and JAERI, and it is also concentrating on producing both ferritic and martensitic steels [49]. The JNC program has been in existence since 1987, and the early work concentrated on ferritic 12Cr ODS steels. More recently, work has been conducted on a martensitic (9Cr–0.12C–2W–0.20Ti–0.35 $\text{Y}_2\text{O}_3$ ) ODS steel. A 12Cr–0.3C–2W–0.30Ti–0.23  $\text{Y}_2\text{O}_3$  steel produced extremely high creep strength in the longitudinal (axial) direction, but with a considerably lower strength in the hoop direction (Fig. 8). The martensitic steel, on the other hand, had a lower creep strength, but without the anisotropy (Fig. 8).

At Kyoto University the objective is to develop a martensitic ODS steel based on the JLF-1 composition and also develop a 12Cr ferritic ODS steel. Work is concentrated on optimizing processing procedures and

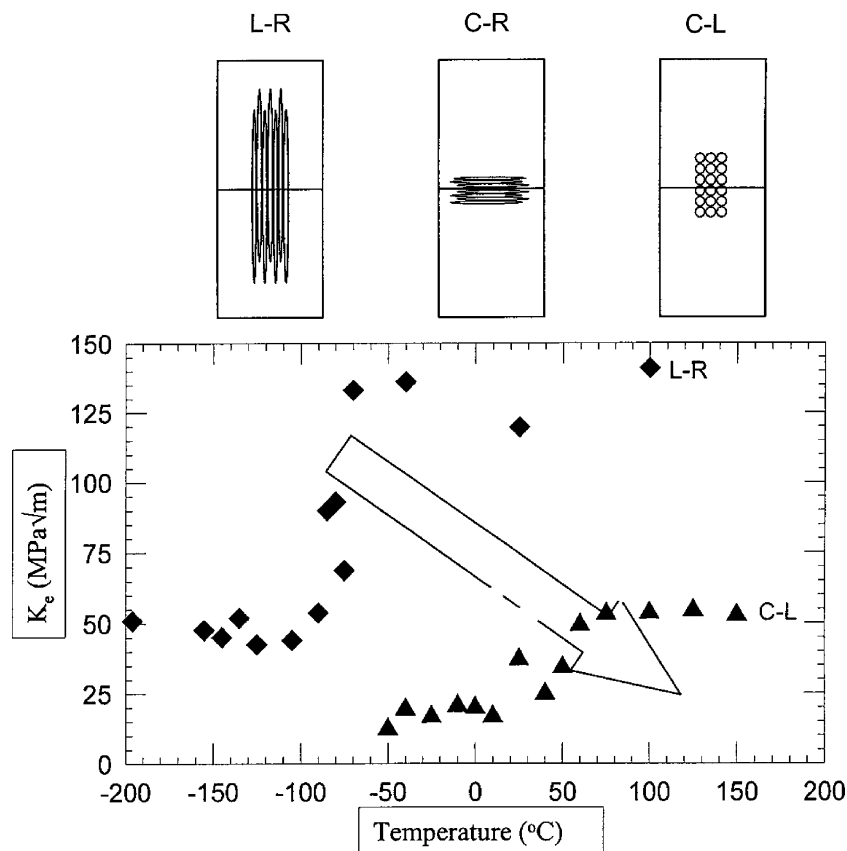


Fig. 6. The effect of orientation on the toughness of a MA957 rod [44].

heat treatments, evaluating mechanical properties and thermal stability, and proceeding with irradiation experiments [49]. Finally, JAERI is developing ODS martensitic steels based on the F82H composition. The initial work illustrates the problems with ODS steels. Although room temperature yield stress of some experimental steels was almost 100% greater than that of F82H, the impact properties, as would be expected, were degraded, with the transition temperature 100–160 °C higher than that of F82H [49].

At ORNL, TEM examination of an Fe–12.3Cr–3W–0.39Ti–0.25Y<sub>2</sub>O<sub>3</sub> (designated 12CrYWTi) and an Fe–12.4Cr–0.25 Y<sub>2</sub>O<sub>3</sub> (12CrY1) ODS steel produced by Kobe Steel Ltd. indicated that the 12CrYWTi contained 3–5 nm diameter particles at a number density of  $1\text{--}2 \times 10^{23} \text{ m}^{-3}$ , whereas the 12CrY1 contained 10–40 nm particles at a density of  $10^{20}\text{--}10^{21} \text{ m}^{-3}$  [50]. Electron diffraction indicated that particles in the 12CrY1 were essentially pure Y<sub>2</sub>O<sub>3</sub> [50]. Because of the size of the particles in 12CrYWTi, three-dimensional atom probe (3-DAP) was used to determine that the particles were atom clusters enriched in Y, Ti and O. The original Y<sub>2</sub>O<sub>3</sub> particles evidently dissolved during processing,

and new clusters formed, in which the concentration of titanium was higher than the yttrium [51].

To determine the effect of tungsten, an Fe–12.4Cr–3W–0.25Y<sub>2</sub>O<sub>3</sub> (12CrYW) steel was examined with the 3-DAP and compared with the 12CrYWTi [52]. The clusters in the 12CrYW, like those in 12CrY1, were highly enriched in Y and O, while those in 12CrYWTi were highly enriched in Y, Ti and O. With the 3-DAP, the particle densities were estimated at  $1.4 \times 10^{24}$  (an order of magnitude larger than determined by TEM [50]) and  $3.9 \times 10^{23} \text{ m}^{-3}$  for the 12CrYWTi and 12CrYW, respectively, with average Guinier radii estimated at  $2.0 \pm 0.9$  and  $2.4 \pm 0.8$  nm, respectively. Therefore the presence of the tungsten in the absence of titanium apparently increased the number density and reduced the size of the clusters relative to the 12CrY1, and the addition of titanium to the 12CrYW composition caused a further increase in the number density and a decrease in the cluster size [52].

An indication of the potential of ODS steels is illustrated in Fig. 9, where a creep curve is shown for the 12CrYWTi tested at 800 °C in air at 138 MPa [53], and it is compared with a curve for the V–4Cr–4Ti alloy [54],

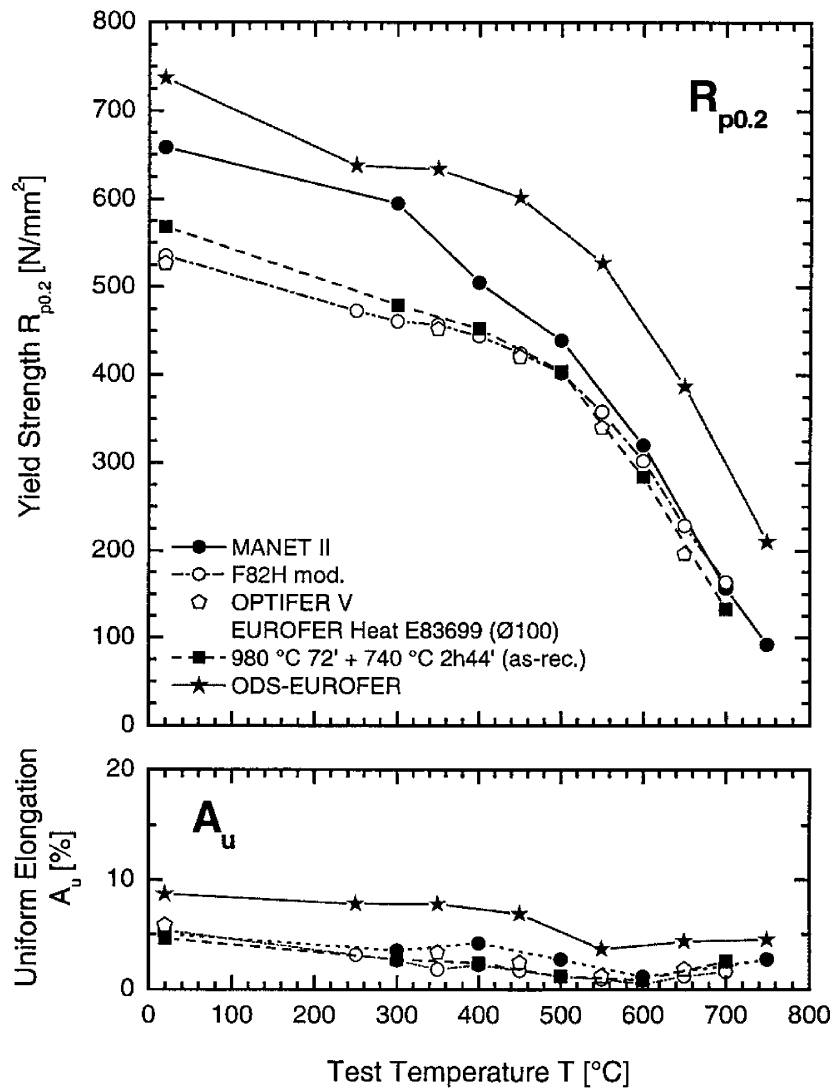


Fig. 7. The 0.2% yield strength and uniform elongation of the EUROFER ODS steel compared to other reduced-activation and conventional ferritic/martensitic steels [48].

another candidate for fusion applications above 600 °C. The V-4Cr-4Ti was tested at 800 °C in vacuum but at a much lower stress of 77 MPa (the V-4Cr-4Ti alloy test is from a pressurized tube specimen and the 12CrYWTi is from a uniaxial specimen). Whereas the vanadium alloy is in tertiary creep prior to rupture (the specimen failed after 4029 h and ~52%), the 12CrYWTi steel at ~5000 h is in the steady-state stage. This specimen ruptured after ~14 500 h (800 °C is an extremely high temperature for a steel with only 12% Cr) with an elongation of ~2.3%, but it failed without a tertiary creep stage.

Thus, considerable progress is being made on the ODS steels. However, it needs to be emphasized that the development on the ODS steels for fusion is still at an

early stage, probably similar to the vanadium alloys. Neither of these materials is at a commercial stage of development comparable to conventional and reduced-activation ferritic/martensitic steels, for which the technology, (i.e., steel production, processing, fabrication, welding, etc.) is advanced to the point that a fusion plant could be constructed. Finally, there is the question of irradiation resistance of ODS steels, for which very little information is available.

## 7. Summary

Ferritic/martensitic steels are at present the leading candidates for a DEMO or fusion power plant. An

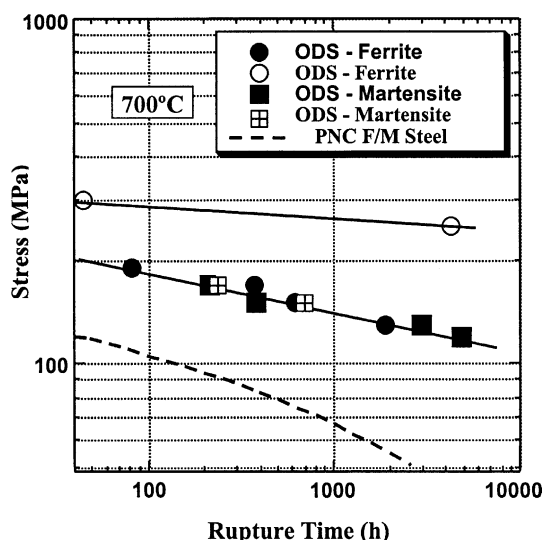


Fig. 8. Creep-rupture strength of ferritic and martensitic ODS steels [49].

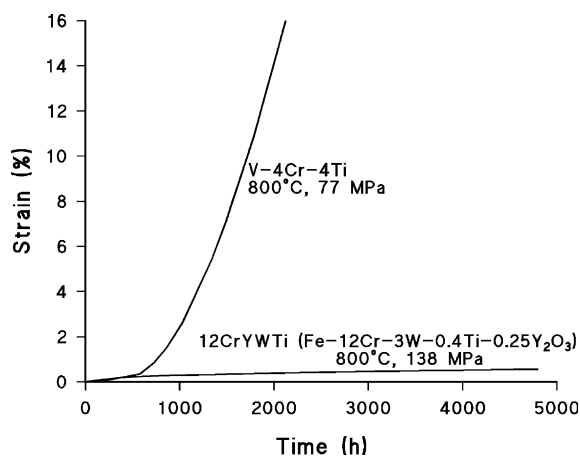


Fig. 9. Comparison of the creep behavior at 800 °C of the 12CrYWTi ODS steel and the V-4Cr-4Ti alloy [53].

international collaboration to determine the feasibility of using the steels for this application has produced a broad range of mechanical and physical property measurements on large heats of reduced-activation steels in the unirradiated and irradiated condition. Uncertainty still exists on the effect of transmutation helium on irradiated properties, a problem that is difficult to address in the absence of a 14 MeV neutron source. Based on the work to date, a large heat of EUROFER 97, a new steel in the European program, was produced and is being investigated. Programs are in progress to develop reduced-activation ferritic/martensitic steels with im-

proved elevated-temperature properties, so that the operating efficiency of fusion reactors can be increased by operating at higher temperatures. Oxide dispersion-strengthened steels offer the best chance of producing a material with enhanced elevated-temperature strength that maintains many of the advantages of the ferritic/martensitic steels.

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### References

- [1] S.N. Rosenwasser, P. Miller, J.A. Dalessandro, J.M. Rawls, W.E. Toffolo, W. Chen, J. Nucl. Mater. 85&86 (1979) 177.
- [2] D.R. Harries, in: J.W. Davis, D.J. Michel (Eds.), Proceedings Topical Conference on Ferritic Alloys for use in Nuclear Energy Technologies, The Metallurgical Society of AIME, Warrendale, PA, 1984.
- [3] H. Attaya, K.Y. Yuan, W.G. Wolfer, G.L. Kulcinski, in: J.W. Davis, D.J. Michel (Eds.), Proceedings of Topical Conference on Ferritic Steels for use in Nuclear Energy Technologies, The Metallurgical Society of AIME, Warrendale, PA, 1984, p. 169.
- [4] T. Lechtenberg, C. Dahms, H. Attaya, in: J.W. Davis, D.J. Michel (Eds.), Proceedings of Topical Conference on Ferritic Steels for use in Nuclear Energy Technologies, The Metallurgical Society of AIME, Warrendale, PA, 1984, p. 179.
- [5] T. Takagi, J. Tani, P. Ruatto, B. Krevet, L.V. Boccaccini, IEEE Trans. Magn. 32 (3) (1996) 1054.
- [6] L.V. Boccaccini, P. Ruatto, Fusion Technol. (1997) 1519.
- [7] M. Sato, Y. Miura, S. Takeji, H. Kimura, K. Shiba, J. Nucl. Mater. 258–263 (1998) 1253.
- [8] T. Nakayama, M. Abe, T. Tadokoro, M. Otsuka, J. Nucl. Mater. 271&272 (1999) 491.
- [9] S. Jitsukawa, M. Tamura, B. van der Schaaf, R.L. Klueh, A. Alamo, C. Petersen, M. Schirra, G.R. Odette, F. Tavassoli, K. Shiba, A. Kohyama, A. Kimura, J. Nucl. Mater., this conference, these Proceedings.
- [10] K. Shiba, T. Sawai, Y. Kohno, A. Kohyama, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 151.
- [11] M.A. Sokolov, K. Shiba, R.L. Klueh, G.R. Odette, J. Nucl. Mater., in press.
- [12] R.L. Klueh, M.A. Sokolov, K. Shiba, Y. Miwa, J.P. Robertson, J. Nucl. Mater. 283–287 (2000) 478.
- [13] R. Lindau, A. Möslang, D. Preininger, M. Rieth, H.D. Röhrig, J. Nucl. Mater. 271&272 (1999) 450.
- [14] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 187 (1992) 60.
- [15] E.V. van Osch, M.G. Horsten, G.E. Lucas, G.R. Odette, in: M.L. Hamilton, A.S. Kumar, S.T. Rosinski, M.L.

- Grossbeck (Eds.), Effects of Irradiation on Materials: 19th International Symposium, ASTM STP 1366, American Society for Testing and Materials, West Conshohocken, PA, 2000, p. 612.
- [16] E.I. Materna-Morris, M. Rieth, K. Ehrlich, in: M.L. Hamilton, A.S. Kumar, S.T. Rosinski, M.L. Grossbeck (Eds.), Effects of Irradiation on Materials: 19th International Symposium, ASTM STP 1366, American Society for Testing and Materials, West Conshohocken, PA, 2000, p. 597.
- [17] K. Shiba, J. Nucl. Mater. 283–287 (2000) 474.
- [18] E. Wakai, N. Hashimoto, Y. Miwa, J.P. Robertson, R.L. Klueh, K. Shiba, S. Jitsukawa, J. Nucl. Mater. 283–287 (2000) 799.
- [19] Y. Miwa, E. Wakai, K. Shiba, N. Hashimoto, J.P. Robertson, A.F. Rowcliffe, A. Hishinuma, J. Nucl. Mater. 283–287 (2000) 334.
- [20] T. Morimura, A. Kimura, H. Matsui, J. Nucl. Mater. 239 (1996) 118.
- [21] D.S. Gelles, M.L. Hamilton, G.L. Hankin, J. Nucl. Mater. 251 (1997) 188.
- [22] R. Kasada, A. Kimura, H. Matsui, M. Narui, J. Nucl. Mater. 258–263 (1998) 1199.
- [23] A. Kimura et al., these Proceedings.
- [24] P.J. Maziasz, R.L. Klueh, J.M. Vitek, J. Nucl. Mater. 141–143 (1986) 929.
- [25] D.S. Gelles, S. Ohnuki, K. Shiba, A. Kohyama, J.P. Robertson, M.L. Hamilton, Fusion Materials Semiannual Progress Report for the Period Ending 31 December 1998, US Department of Energy, DOE/ER-0313/25, April 1999, p. 143.
- [26] D.S. Gelles, M.L. Hamilton, B.M. Oliver, L.R. Greenwood, Fusion Materials Semiannual Progress Report for the Period Ending 31 December, 1998, US Department of Energy, DOE/ER-0313/27, March 2000, p. 149.
- [27] D.S. Gelles, M.L. Hamilton, B.M. Oliver, L.R. Greenwood, Fusion Materials Semiannual Progress Report for the Period Ending 31 December 1998, US Department of Energy, DOE/ER-0313/29, April 2001, p. 93.
- [28] L.R. Greenwood, B.M. Oliver, S. Ohnuki, K. Shiba, Y. Kohno, A. Kohyama, J.P. Robertson, J.W. Meadows, D.S. Gelles, J. Nucl. Mater. 283–287 (2000) 1438.
- [29] R. Kasada, T. Morimura, A. Hasegawa, A. Kimura, J. Nucl. Mater. 299 (2001) 83.
- [30] M. Eldrup, B.N. Singh, M. Victoria, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 402.
- [31] K.K. Bae, K. Ehrlich, A. Möslang, J. Nucl. Mater. 191–194 (1992) 905.
- [32] X. Jia, Y. Dai, G. Bauer, R. Schaeublin, M. Victoria, J. Nucl. Mater., in press.
- [33] B. van der Schaaf, D.S. Gelles, S. Jitsukawa, A. Kimura, R.L. Klueh, A. Möslang, G.R. Odette, J. Nucl. Mater. 283–287 (2000) 52.
- [34] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, J. Nucl. Mater. 233–237 (1996) 138.
- [35] Fusion Technology: Annual Report of the Association CEA/EURATOM, 2000, p. 199.
- [36] Nuclear Fusion Programme Annual Report of the Association Forschungszentrum Karlsruhe/EURATOM, October 1999–September 2000, Forschungszentrum Karlsruhe, FZKA 6550 and EUR 19707 EN, December 2000, p. 138.
- [37] Fusion Technology: Annual Report of the Association CEA/EURATOM, 2000, p. 203.
- [38] F. Masuyama, in: R. Viswanathan, J.W. Nutting (Eds.), Advanced Heat Resistant Steels for Power Generation, IOM Communications, London, 1999, p. 33.
- [39] H. Sakasegawa, T. Hirose, T. Suzuki, A. Kohyama, Y. Katoh, T. Harada, K. Asakura, T. Kumagai, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 494.
- [40] Y. Kohno, T. Hirose, A. Kohyama, A. Kimura, K. Asakura, M. Narui, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 476.
- [41] S. Ukai, T. Yoshitake, S. Mizuta, Y. Matsudaira, S. Hagi, T. Kobayashi, J. Nucl. Sci. Technol. 36 (1999) 710.
- [42] M.L. Hamilton, D.S. Gelles, R.J. Lobsinger, G.D. Johnson, W.F. Brown, M.M. Paxton, R.J. Puigh, C.R. Eiholzer, C. Martinez, M.A. Blotter, Oxide Dispersion Strengthened Alloy MA957 for Fast Reactor Applications, Pacific Northwest National Laboratory, PNNL-13168, February 2000.
- [43] D.K. Mukhopadhyay, F.H. Froes, D.S. Gelles, J. Nucl. Mater. 258–263 (1998) 1209.
- [44] M.J. Alinger, G.R. Odette, G.E. Lucas, D.S. Gelles, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 235.
- [45] Fusion Technology: Annual Report of the Association CEA/EURATOM, 2000, p. 215.
- [46] Fusion Technology: Annual Report of the Association CEA/EURATOM, 2000, p. 243.
- [47] Fusion Technology: Annual Report of the Association CEA/EURATOM, 2000, p. 321.
- [48] K. Ehrlich, R. Lindau, A. Möslang, M. Schirra, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 305.
- [49] S. Ukai, A. Kohyama, A. Kimura, T. Hirose, K. Shiba, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 282.
- [50] I.-S. Kim, J.D. Hunn, N. Hashimoto, D.J. Larson, P.J. Maziasz, K. Miyahara, E.H. Lee, J. Nucl. Mater. 280 (2000) 264.
- [51] D.J. Larson, P.J. Maziasz, I.-S. Kim, K. Miyahara, Scrip. Mater. 44 (2001) 359.
- [52] M.K. Miller, E.A. Kenik, K.F. Russell, L. Heatherly, D.T. Hoelzer, P.J. Maziasz, Mater. Sci. Eng. A, in press.
- [53] R.L. Klueh, P.J. Maziasz, I.S. Kim, L. Heatherly, D.T. Hoelzer, N. Hashimoto, E.A. Kenik, K. Miyahara, J. Nucl. Mater., these Proceedings.
- [54] R.J. Kurtz, M.L. Hamilton, in: Fusion Materials Semiannual Progress Report for Period Ending 30 June 1999, DOE/ER-0313/26, September 1999, p. 3.