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Assessment of Impact Test Experiments on Irradiated EUROFER97 and other RAFM Steels

Final Report for Task TW5-TTMS 001-D11

E. Gaganidze

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Bewertung von Kerbschlagbiegeuntersuchungen an bestrahlten EUROFER97 und anderen RAFM Stählen

Zusammenfassung

Der vorliegende Bericht fasst die Bewertung von Kerbschlagbiegeuntersuchungen zusammen, die weltweit an unterschiedlichen Institutionen am bestrahlten europäischen niedrigaktivierbaren Stahl EUROFER97 sowie an weiteren bestrahlten niedrigaktivierbaren Stählen (F82H, OPTIFER) durchgeführt wurden. Die Arbeit wurde im Rahmen des europäischen Abkommens zur Entwicklung der Fusionsforschung (EFDA), Subtask TW5-TTMS-001-D11, vorgenommen. Die Auswertung der Kerbschlagbiegentersuchungen zeigt bei Bestrahlungstemperaturen unterhalb von $T_{irr} \leq 330$ °C eine zunehmende Versprödung von EUROFER97 mit zunehmender Bestrahlungsdosis (bis 32 dpa). Austenisierung von EUROFER97 bei einer höheren Temperatur (1040 °C) im Vergleich zu Anlieferzustand führt zur Versprödungsabnahme bei Bestrahlungstemperaturen unterhalb von $T_{irr} \leq 330$ °C. Internationale RAFM (F82H, OPTIFER) Stähle zeigen ein mit dem EUROFER97 vergleichbares Bestrahlungsverhalten. Die beobachtete deutliche Korrelation zwischen der bestrahlungsinduzierten Versprödung und der Verfestigung niedrigaktivierbarer Stähle bei Bestrahlungstemperaturen unterhalb von $T_{irr} \leq 350$ °C deutet auf den Verlagerungsschädigungsmechanismus der Tieftemperaturversprödung. EUROFER97 und weitere niedrigaktivierbare Stähle zeigen eine ausreichende Resistenz gegen Neutronenbestrahlung bei Bestrahlungstemperaturen oberhalb von $T_{irr} \geq 350$ °C bis zu 16.3 dpa.

Ein Einfluss der Bohr-Helium-Umwandlung auf Versprödung von niedrigaktivierbaren Stählen ist bei 16.3 dpa @300 °C Bestrahlung für den Bohr-Gehalt zwischen 4 und 62 wppm nicht erkennbar. Bohrdotierte Proben (Bohrgehalt zwischen 82-1120 wppm) zeigen dagegen zunehmende Versprödung und eine Abnahme der Zähigkeit bei Zunahme des generierten Heliumgehalts. Bei einem Heliumgehalt von 84 appm ist die Helium induzierte Versprödung auf Helium induzierte Verfestigung zurückzuführen. Höhere Heliumkonzentrationen führen neben Helium induzierter Verfestigung zu weiteren Versprödungsmechanismen.

Abstract

This report summarizes assessment of Charpy impact experiments carried out by different worldwide institutions on irradiated European RAFM steel EUROFER97 and other RAFM steels (F82H, OPTIFER). The assessment was carried out under the contract of the European Fusion Development Agreement (EFDA), subtask TW5-TTMS-001-D11. Irradiation resistance of EUROFER97 is not satisfactory at low irradiation temperatures $T_{irr} \leq 330$ °C, *i.e.* large, non-saturating low temperature embrittlement is seen up to 32 dpa. Heat treatment of EUROFER97 at higher austenization temperature (1040°C) leads to the reduction of the embrittlement at low irradiation temperatures $T_{irr} \leq 330$ °C. Irradiation performance of EUROFER97 is comparable to that of international RAFM (F82H, OPTIFER) steels. A close correlation between irradiation induced embrittlement and hardening found at $T_{irr} \leq 350$ °C for RAFM steels indicates displacement damage nature of low temperature embrittlement. Irradiation performance of EUROFER97 and international reference RAFM steels is satisfactory at $T_{irr} \geq 350$ °C up to 16.3 dpa.

The influence of boron-to-helium transformation on embrittlement cannot be resolved for 16.3 dpa irradiated RAFM steels with boron contents between 4 and 62 wppm. Boron doped steels (boron content between 82-1120 wppm) show progressive embrittlement and reduction of toughness with increasing helium amount. He induced embrittlement is of hardening nature for helium content of 84 appm. Larger helium concentrations lead to non-hardening embrittlement mechanisms beyond that of hardening embrittlement.

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FIGURES

1 Objective

Charpy impact testing on KLST type specimens is a widely used testing method for characterization of the structural material irradiation resistance. This led to the development of a large data base on the ductile-to-brittle transition temperature (DBTT). Although the reduced activation ferritic martensitic (RAFM) 7-10%CrWVTa steels (EUROFER, OPTIFER, F82H) exhibits clearly better irradiation performance compared to the modified commercial 10-11%-Cr-NiMoVNb steels, the hardening induced by neutron irradiation accompanied by the embrittlement and by reduction of toughness still remain a main concern. He and H generation is another important issue that requires careful investigation prior to structural material qualification. The assessment of impact properties of irradiated RAFM steels (EUROFER, F82H, OPTIFER) will support material database generation and specification of new EUROFER charge. The study of correlation of fracture mechanical and impact testing results on irradiated RAFM steels is an another important issue thoroughly discussed in [1].

2 Data Collection

Data have been collected from associations having performed irradiated impact tests on RAFM steels, or retrieved by existing literature and databases.

2.1 Materials

EUROFER97

An industrial batch of the European RAFM steel EUROFER97 (nominal composition Fe-9Cr-1.1W-0.2V-0.12Ta) was produced by Böhler Austria GmbH. Four different product forms: plates, with thickness of 8, 14, 25 mm and bars with diameter of 100 mm have been distributed by FZK to different European associations. Normalization was performed at 980 °C/0.5 h and tempering, followed by air cooling was done at 760 °C/1.5 h for the plates and at 740 °C/3.7 h for the bars.

F82H

A 5-ton heat of standard F82H (F82H-Std, nominal composition Fe-7.5Cr-2W-0.2V-0.04Ta-0.0034B-0.1C) was produced by NKK Corporation, Kawasaki, Japan by order of the Japan Atomic Energy Research Institute (JAERI). A 5-ton heat of modified F82H (F82H-mod, Fe-7.5Cr-2W-0.15V-0.02Ta-0.1C) was also produced by NKK Corporation for collaborative research coordinated by an International Energy Agency (IEA) committee. The 7.5 mm, 15 mm and 25 mm plates have been distributed by the IEA, and subsequently by FZK to the European partners. The plate materials have been tested in the as-received and in different optimum heat-treated conditions.

Other RAFM Steels

FZK has been intensively involved in the development of the reduced activated ferritic/martensitic steels via systematic variation of alloying elements. OPTIFER family steels are 9.5% Cr-MnVTa steels with 1% W or W-free variants with germanium. He embrittlement is investigated in different structural steels as well as in EUROFER97 based experimental heats, that are doped with different contents of natural boron and the separated ^{10}B-isotope (0.008-0.112 wt.%).

An experimental heat of ORNL 9Cr-2WVTa was produced for Oak Ridge National Laboratory. The button melts were rolled to 6.4 mm plates. The plates were normalized at 1050°C/1h and tempered at 750°C/1h.

NRG 9Cr1WVTa EUROFER lab heat was ordered by NRG at Corus England, then British steel, as a 50 kg batch. The specification was identical to that of EUROFER97. The ingot was formed as a forged bar, later hot rolled to 25 mm plate. Another NRG 9Cr2WVTa experimental lab heat was manufactured in 1996.

2.2 Impact Testing at FZK

Impact properties of EUROFER97 and other RAFM (F82H, F82H-mod, OPTIFER series, GA3X) steels were intensively studied at FZK in different irradiation programmes (MANITU, HFR Phase Ia, Ib, IIa, IIb (SPICE), ARBOR I) up to 32.8 dpa [2],[3],[4],[5],[6],[7],[8][9],[10]. Reduced sized Charpy V specimens of KLST type (3x4x27 mm^3) have been used for quantification of neutron irradiation induced material embrittlement and hardening in a wide irradiation temperature range of 250-450 °C. The irradiation performance of EUROFER97 was studied in the as-received condition (EUROFER97 ANL: 980 °C/0.5 h + 760 °C/1.5 h) and after heat treatment at higher austenitizing temperature (EUROFER97 WB: 1040 °C/0.5 h + 760 °C/1.5 h). Table 6-1 summarises impact properties of KLST specimens. A thorough investigation of the reference unirradiated state of EUROFER97 for different heat treatment conditions and for different product forms was performed with standard ISO-V Charpy (10x10x55 mm^3) specimens in [11] and is summarized in Table 6-2. Impact testing of 70 dpa irradiated Charpy-V KLST specimens from ARBOR II is expected at the end of year 2007.

2.3 Impact Testing at SCK·CEN

Within IRFUMA I, II and III irradiation campaigns SCK·CEN investigated fracture toughness and impact properties of EUROFER97 irradiated up to 0.25, 1.0 and 2.25 dpa nominal doses at 300 °C [12]. Specimens have been fabricated from the ∅=100 mm bars.

Impact tests were done on standard Charpy V-notch specimens (ISO-V: 10x10x55 mm^3). In order to increase the number of available data points, several Charpy specimens have been obtained by reconstituting previously broken samples. Taking into account individual fluence values, the following sub-groups were considered for analyses of the impact test results: Sub-group 1 (0.34±0.066 dpa), Sub-group 2 (0.71±0.167 dpa) and Sub-group 3 (1.55±0.313 dpa). The results on the unirradiated and irradiated specimens are summarized in Table 6-3. A progressive material embrittlement is seen with increasing irradiation dose.

2.4 Impact Testing at NRG

Impact properties on unirradiated and up to 8.9 dpa irradiated EUROFER97 and other RAFM steels have been thoroughly studied by NRG in the framework of different irradiation programs (SUMO-02÷SUMO-07, SIWAS-06, SIWAS-07, SIWAS-09, SINAS-80/6, SINAS-80/7, CHARIOT-02, CHARIOT-04) [13],[14]. Irradiation was performed at 300 °C and 60 °C. EUROFER97 specimens have been machined from 8, 14 and 25 mm plates. The results of KLST impact tests on unirradiated and irradiated EUROFER97 and other RAFM steels are summarised in Table 6-4.

2.5 Impact Testing at ORNL

ORNL intensively studied impact properties of several RAFM steels [15]. F82H, F82H-mod, F82H with 2% Ni, 9Cr-2WVTa steels have been irradiated in the ORNL High Flux Isotope Reactor (HFIR). Impact test were performed on miniaturised, one third size V-notch specimens (3.3x3.3x25.4 mm^3) with a 0.51 mm deep 30° V-notch and a 0.05-0.08 mm root radius.

Table 6-5 shows the DBTT, the shift in the DBTT and the USE of F82H IEA heats in the as received and heat treated (HT) conditions. The specimens were machined in the L-T orientation and subsequently irradiated at 300, 380 and 500 °C target temperatures. An additional HT to reduce prior austenite grain size (from 125 to 55 µm) resulted in slightly better initial properties (lower DBTT and larger USE) compared to the as received condition but led to no observable effect in the shift in the DBTT.

3 Data Assessment

3.1 EUROFER97

Unirradiated State

For ISO-V specimens machined from the 14 mm EUROFER97 plate no significant differences in DBTT were observed between the as delivered (980 °C+760 °C) and heat treated (1050 °C+750 °C) conditions, see Table 6-2. After austenitizing at 1075 °C, however, the ISO-V DBTT shifts to a higher temperature, independently from the specimen orientation (transverse or longitudinal). ISO-V specimens machined from the ∅ 100 mm bar exhibited as expected significant difference in DBTT between transverse and longitudinal orientations.

Fig. 3-1 shows KLST impact data on unirradiated EUROFER 97 from [9],[13]. The specimens were machined in L-T orientations from plates of different thicknesses of 8, 14 and 25 mm. There is a considerable scattering in the DBTT between -90 and -68 °C. Even the specimens machined from the 25 mm thick plate of the same heat (83697) show remarkably different DBTTs of -68 °C and -81 °C for NRG and FZK experiments, respectively. The DBTT from latter experiment is closer to the DBTT of NRG experiment for 8 mm plate.

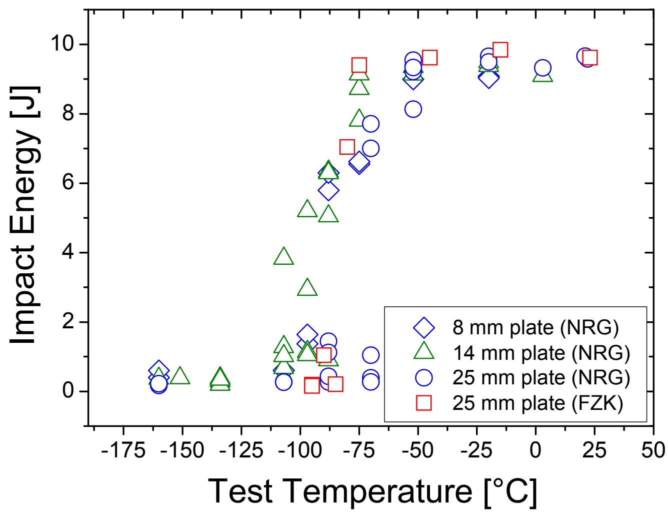

Fig. 3-1 KLST Impact energy vs. test temperature curves on unirradiated EUROFER 97. The thicknesses of plates along with data sources are indicated in legend, see [9],[13].

Irradiated State (T_{irr} =300-330 °C)

Fig. 3-2 shows KLST impact data on unirradiated and irradiated 25 mm thick EUROFER97 plate. NRG data show progressive embrittlement with irradiation dose accompanied by decrease of the upper shelf energy (USE) – behaviour typical for RAFM steels (OPTIFER, F82H). The data scattering increases for 2.4 dpa/300 °C and 8.9 dpa/300 °C irradiation condition partly due to different doses received by individual specimens. Remarkably, 14.8 dpa/300 °C FZK data show lower embrittlement and the narrower ductile-to-brittle transition region than 8.9 dpa/300 °C NRG data, though the USEs are comparable. 15 dpa/330 °C WTZ data show impact properties that resemble to those of 14.8 dpa/300 °C FZK data in the transition region. The lower DBTT for 15 dpa/330 °C WTZ data (95°C) compared to 14.8 dpa/300 °C FZK data (106 °C) can be prescribed to the higher irradiation temperature in the former case. 31.8 dpa/332 °C FZK data show further embrittlement, though the USE is comparable to those of 14.8 dpa/300 °C FZK and 15 dpa/330 °C WTZ data.

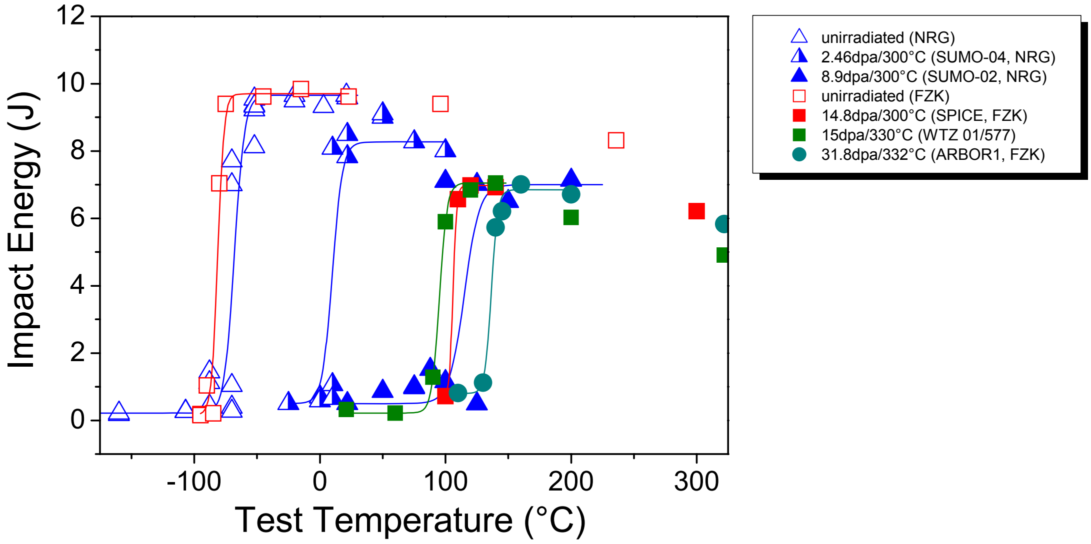

Fig. 3-2 KLST Impact energy vs. test temperature curves on unirradiated and irradiated EUROFER 97. The specimens are machined from the as-delivered 25 mm plate (heat 83697). Irradiation conditions are indicated in legend, see [9],[13].

Fig. 3-3 shows neutron irradiation induced embrittlement vs. irradiation dose for EUROFER97 for irradiation temperatures (T_{irr}) between 300 and 330 °C [9],[12],[13]. The DBTT shifts are quantified by impact tests on KLST and on ISO-V Charpy specimens. The results of impact testing with respect of material embrittlement are consistent for KLST and ISO-V specimens within typical experimental uncertainties in determination of DBTT. No embrittlement saturation can be observed up to 32 dpa. Large data scattering observed at intermediate doses prevents an unambiguous quantitative description of the embrittlement trend. The line is a least square fit to the impact DBTT with a function of type

$$\Delta T_0 = A\left[1-\exp\left(-\frac{dose}{\tau}\right)\right] \quad . \qquad (1)$$

The best fit was obtained by weighting the data with respect to irradiation dose (weighting factors $\propto$ Dose) and with the fitting parameters A=217.23 °C and τ=7.15 dpa.

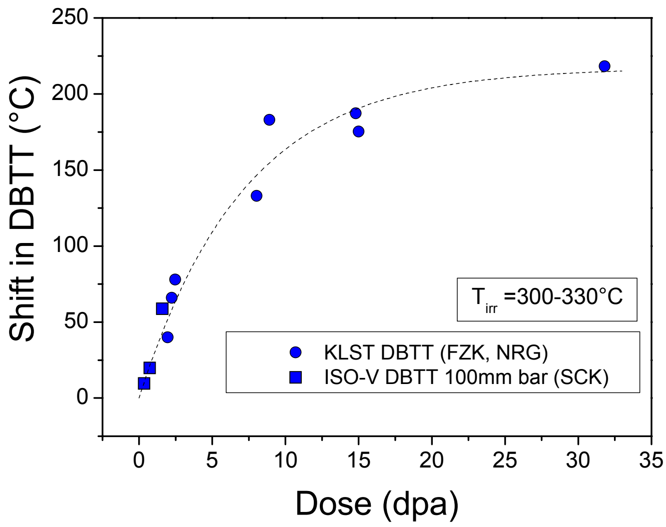

Fig. 3-3 Irradiation induced shift in DBTT vs. irradiation dose assessed for EUROFER97 [9],[12],[13]. The line is a least square fit to DBTT data with Eq. (1) using weighting with respect to irradiation dose (weighting factors $\propto$ Dose). A=217.23 °C and τ=7.15 dpa.

3.2 F82H

Fig. 3-4 shows neutron irradiation induced embrittlement vs. irradiation dose for F82H family steels for irradiation temperatures (T_{irr}) between 300 and 330 °C [2]-[10],[13]-[15]. The DBTT shifts are quantified by impact tests on KLST and on one third size Charpy-V specimens. No embrittlement saturation can be observed up to 32 dpa as was the case for EUROFER97 in Fig. 3-3. The data scattering for the low and intermediate irradiation doses, i.e. between 2.4 and 10 dpa, however, is more pronounced for F82H family steels than for EUROFER97. This is partly explained by the fact that, besides uncertainties in the irradiation doses and/or irradiation temperatures seen by individual specimens, for F82H additional variations in material properties exists due to variations in the heat compositions and variations in fabrication routes. This large data scattering observed at low and intermediate doses prevents an unambiguous quantitative description of the embrittlement trend. The line in Fig. 3-4 is a least square fit to DBTT data with Eq. (1) using data weighting with respect to irradiation dose. The best fit was obtained with fit parameters A=229.83 °C and τ=6.58 dpa.

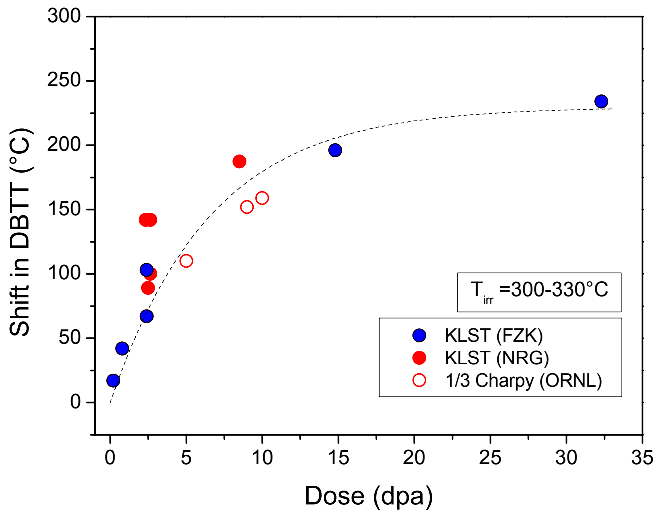

Fig. 3-4 Irradiation induced shift in DBTT vs. irradiation dose assessed for F82H, F82H-mod [2]-[10],[13]-[15]. The line is a least square fit to DBTT data with Eq. (1) using data weighting with respect to irradiation dose (weighting factors ∝ Dose). A=229.83 °C and τ=6.58 dpa.

3.3 Effect of Irradiation Temperature

Fig. 3-5 shows KLST DBTT of selected RAFM steels for different irradiation conditions [2]-[10],[13],[14]. Clearly, RAFM steels exhibit better resistance against neutron irradiation at irradiation temperatures above $T_{irr} \geq 350$ °C than at irradiation temperatures below $T_{irr} \leq 330$ °C.

The role of irradiation temperature on the impact properties was extensively studied by FZK in different irradiation campaigns [2]-[9]. Fig. 3-6 shows the USE as a function of irradiation temperature for RAFM steels from SPICE irradiation campaign for an average damage dose of 16.3 dpa [9]. At low irradiation temperatures ($T_{irr} \leq 300$ °C), the USE of EUROFER97 ANL is strongly affected by neutron irradiation, this effect being most pronounced at 300°C. The USE recovers at higher irradiation temperatures ($T_{irr} \geq 350$ °C), though it still remains below the USE under unirradiated conditions of 9.8 J. The impact toughness properties of EUROFER97 WB are influenced by 250, 350 and 450 °C neutron irradiation in quite similar ways. The reference steel OPTIFER-Ia irradiated at 300 °C shows the highest USE of 8.1 J, while the USE of F82H-mod irradiated at the same temperature is comparable to that of EUROFER97 ANL. MANET-I (non RAFM steel) shows the worst impact toughness after neutron irradiation at 300 °C. The results of OPTIFER-Ia, F82H-mod and GA3X irradiated at 400 °C show USE comparable to that of EUROFER97 ANL. The finest grain structure of OPTIFER-Ia might explain the superior USE of OPTIFER-Ia under unirradiated and irradiated conditions.

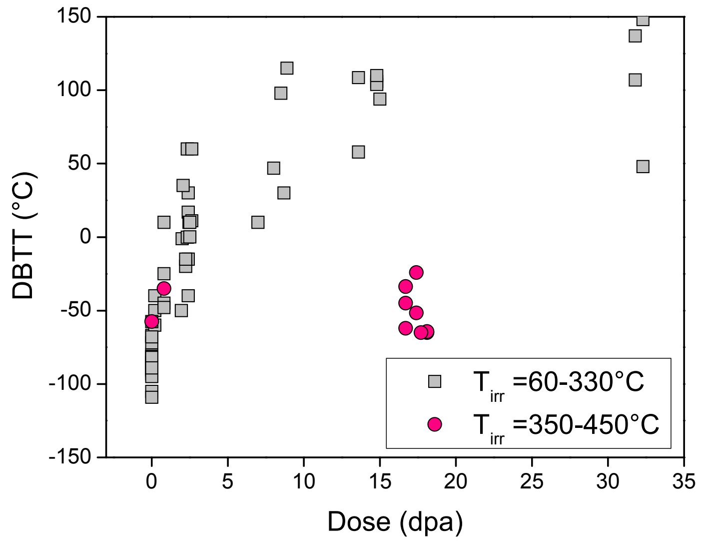

Fig. 3-5 KLST DBTT of RAFM steels (EUROFER97, F82H, F82H-mod, OPTIFER-Ia, OPTIFER-IVc) for different irradiation conditions [2]-[10],[13],[14].

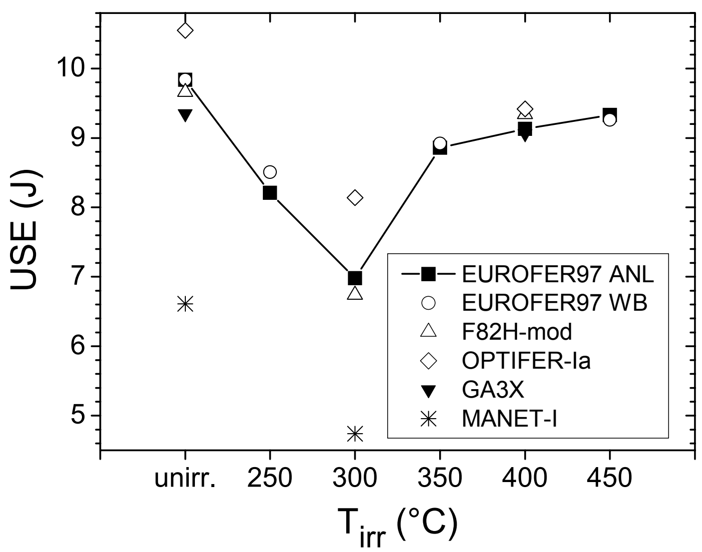

Fig. 3-6 KLST Upper Shelf Energy vs. irradiation temperature for several RAFM steels for an average damage dose of 16.3 dpa (SPICE) [9]. For comparison, the results obtained under unirradiated conditions are included.

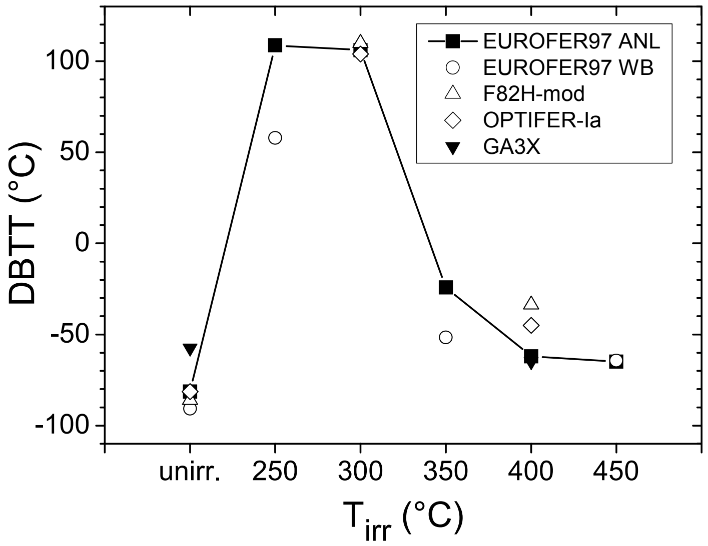

Fig. 3-7 KLST DBTT vs. irradiation temperature for RAFM steels for an average damage dose of 16.3 dpa (SPICE) [9].

Fig. 3-7 shows the DBTT as a function of irradiation temperature for RAFM steels from SPICE irradiation campaign for a damage dose of 16.3 dpa [9]. The DBTT of all materials investigated is influenced most at low irradiation temperatures (T_{irr} ≤ 300 °C). Remarkably, the DBTT of EUROFER97 WB at T_{irr} =250 °C is smaller than the DBTT of EUROFER97 ANL by 50 °C. The difference of the DBTTs of EUROFER97 materials austenitized at different temperatures decreases with increasing irradiation temperature and vanishes completely at 450 °C. The DBTT of reference F82H-mod and OPTIFER-Ia materials is comparable to that of EUROFER97 ANL at T_{irr} =300 °C. However, it has to be mentioned that for F82H-mod, the slope of the curve in the transition region is smaller than the corresponding slopes of EUROFER97 ANL and OPTIFER-Ia specimens. The DBTTs of the materials irradiated above 400 °C remain below -24 °C and, hence, are well below the material application temperature.

3.4 Hardening vs. Embrittlement Behaviour

The hardening behaviour of the irradiated steels is shown in Fig. 3-8, where the dynamic yield stress, σ_{Dy}, is plotted vs. the irradiation temperature [9]. The unirradiated values of σ_{Dy} are also included for comparison. Two test temperatures are used for σ_{Dy} analysis: a) RT and b) 100-120 °C (nominal 100°C). At T_{irr}≤ 300 °C EUROFER97 ANL shows strong hardening ($\Delta\sigma_{Dy}$) mostly pronounced at 16.3 dpa /300 °C. At T_{irr}≥ 350 °C the hardening of EUROFER97 ANL is substantially reduced in clear agreement with observation on RAFM steels at lower irradiation doses up to 2.4 dpa [5]-[7]. EUROFER97 WB and reference RAFM steels show basically the same hardening behaviour with T_{irr}.

The hardening vs. embrittlement behaviour was quantified in terms of the hardening shift coefficient C defined as C=ΔDBTT/$\Delta\sigma_{Dy}$ [9]. Fig. 3-9 displays this parameter for room temperature (C_{RT}) and for 100-120°C (C_{100}). At T_{irr}≤ 350 °C, the coefficient C_{100} varies between 0.17≤ C_{100}≤ 0.53 °C/MPa, which is in good agreement with the analysis of 7-9% Cr RAFM steels yielding C=(0.38±0.18) °C/MPa [16] and indicating that embrittlement is dominated by a hardening mechanism. The coefficient C_{100} tends to increase at T_{irr}= 400 °C. This suggests a non-hardening embrittlement (NHE) mechanism [16].

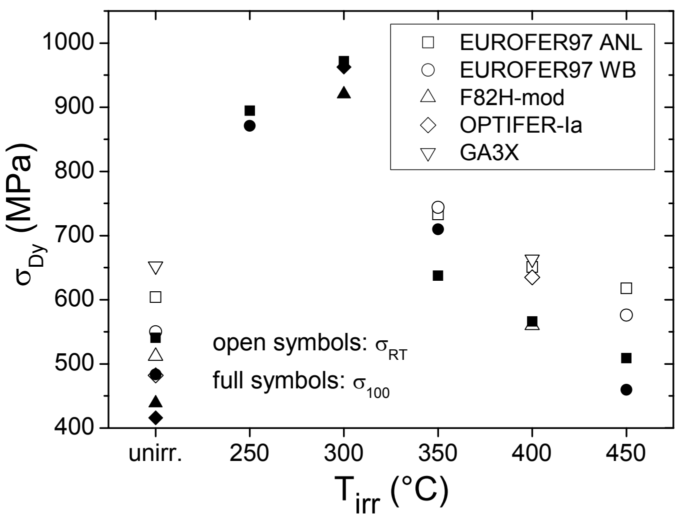

Fig. 3-8 Dynamic yield stress vs. irradiation temperature for RAFM steels for an average damage dose of 16.3 dpa (SPICE); open symbols: σ_{RT}, full symbols σ_{100}, see text for explanation [9].

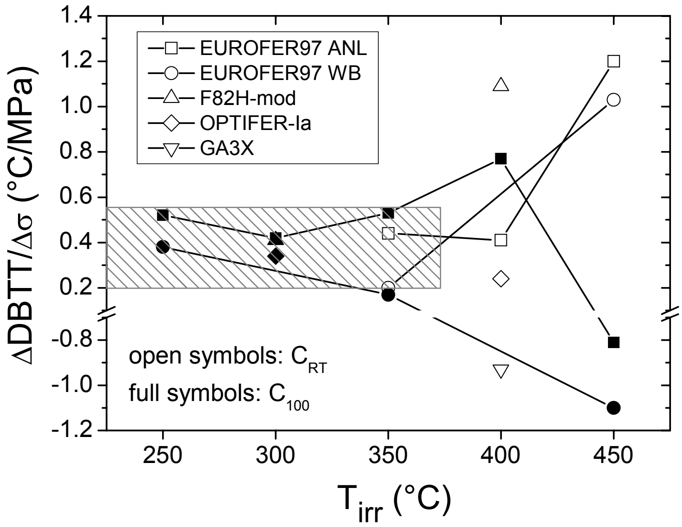

Fig. 3-9 Hardening shift coefficient C vs. irradiation temperature for RAFM steels for an average damage dose of 16.3 dpa (SPICE) [9]; open symbols: C_{RT}, full symbols C_{100}, see text for explanation. The dashed area indicates the scattering band for C from [16].

3.5 Effect of Boron Content

^{10}B isotope is strong absorber of thermal neutrons and transforms to Li and He already at low irradiation doses (after 1.6 dpa already 99.3% of ^{10}B is burn-up). The influence of boron-to-helium transformation on embrittlement cannot be resolved for 16.3 dpa irradiated RAFM steels with boron contents between 4 and 62 wppm, see Fig. 3-7. DBTT of RAFM steels lie between 100-110°C for 16.3 dpa/300 °C irradiation. This is in clear contrast to the results obtained at lower irradiation doses (up to 2.4 dpa), where a close correlation was found between boron content and steel embrittlement [7]. The role of He in a process of embrittlement was investigated by FZK in boron doped steels [8],[17]. Experimental heats ADS2, ADS3 and ADS4 with the basic composition of EUROFER97 were doped with different contents of natural boron and the separated ^{10}B-isotope (natural boron consists of 80% ^{11}B and 20% ^{10}B isotopes). For direct comparison and in order to exclude significant differences in the microstructure ADS2 and ADS3 were doped with 82 wppm nat. B and 83 wppm separated ^{10}B isotope, respectively. ADS4 was doped with 1120 wppm ^{10}B isotope. Helium amounts generated due to ^{10}B burn-up under neutron irradiation are summarized in Table 6-6.

Fig. 3-10 shows Charpy energy vs. test temperature curves for EUROFER97, ADS2 and ADS3 steels. In the unirradiated condition impact properties of the boron doped steels are comparable to those of EUROFER97 indicating negligible chemical effects of boron up to 83 wppm. At T_{irr} =250 °C boron doped steels show up degraded irradiation resistance compared to EUROFER97. Progressive embrittlement and reduction of toughness is seen with increasing helium amount. By considering EUROFER97 as reference, helium induced extra embrittlement (i.e. DBTT shift in boron doped steel after subtraction of DBTT shift in EUROFER97) is estimated in Fig. 3-11. Obviously, embrittlement rate is reduced at higher helium contents. Helium embrittlement can be qualitatively described by a function of type $\propto\{1-\exp(-p_{He}/p_0)\}$, where p_{He} is produced helium amount and p_0 is a fitting parameter.

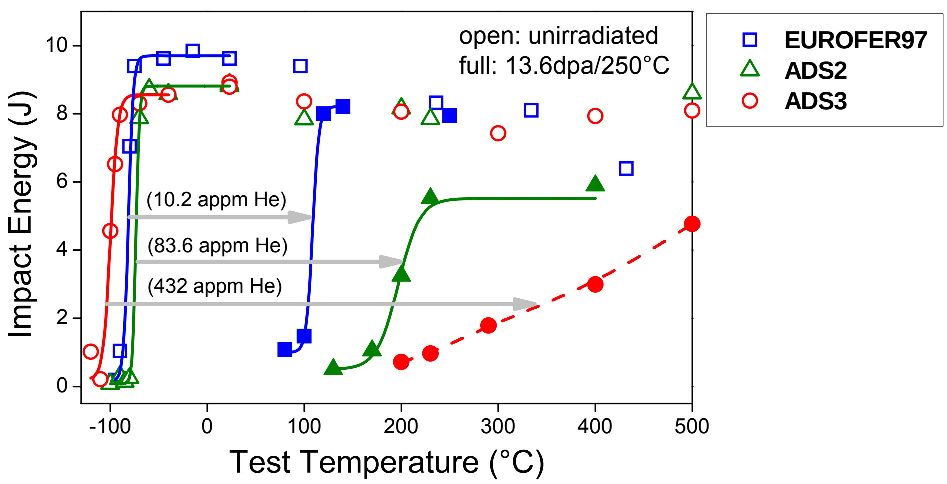

Fig. 3-10 Charpy impact energy vs. test temperature for unirradiated and irradiated EUROFER97, ADS2 and ADS3 steels (irradiation programme SPICE). The arrows indicate the irradiation induced DBTT shifts. Produced helium amounts are indicated in the parentheses.

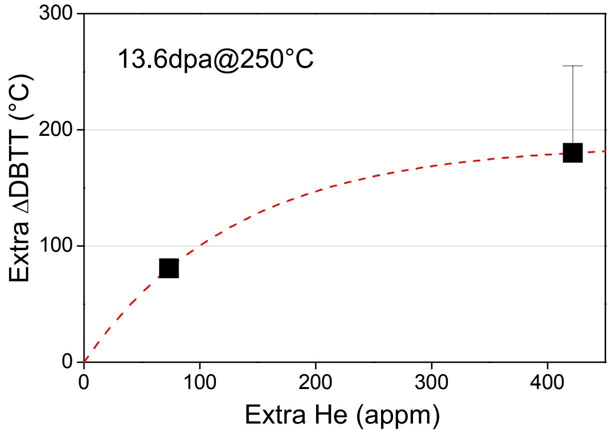

Fig. 3-11 Helium induced extra embrittlement versus extra helium amount (irradiation programme SPICE). Extra $\Delta DBTT=\Delta DBTT_{EUROFER+B}-\Delta DBTT_{EUROFER}$. Extra He = helium amount generated in boron doped heat after subtraction of helium amount generated in EUROFER97. The line is a function of type $\propto\{1-\exp(-p_{He}/p_0)\}$, see text.

The extra embrittlement observed for boron doped steels can not be solely related to produced He amounts. Indeed, OPTIFER-Ia, in Fig. 3-7 shows impact properties comparable to those of EUROFER97 ANL, though in the former case 63.1 appm He is generated due to ^{10}B burn-up. Therefore, not only the He amount but also the evolution of microstructure of the steel during the irradiation is decisive for the irradiation resistance of the steel.

Fig. 3-12 shows the DBTT as a function of irradiation temperature for ADS2 and ADS3 steels. For comparison the DBTT of base EUROFER97 is also included. At all irradiation temperatures ADS3 shows basically larger DBTT than ADS2. Moreover, at T_{irr}=450 °C helium induced extra embrittlement nearly disappears for ADS2, whereas ADS3 shows non-vanishing extra embrittlement. At $T_{irr}\leq$350 °C embrittlement of RAFM steels is mainly of hardening nature and at $T_{irr}\geq$400 °C non-hardening embrittlement mechanisms dominate, see e.g. [9]. At T_{irr}=250 °C the hardening shift coefficient for ADS2 C_{200}=0.57 °C/MPa compares to that of EUROFER97 C_{200}=0.57 °C/MPa indicating that the extra embrittlement of ADS2 is mainly related to extra hardening due to helium production. For ADS3, however, C_{250}=0.74 °C/MPa is much larger than the corresponding value for EUROFER97 C_{250}=0.56 °C/MPa indicating the existence of additional embrittlement mechanisms beyond to the helium induced hardening. At T_{irr}=450°C the hardening shift coefficient C_{RT} was 0.64 and 1.08 °C/MPa for ADS2 and ADS3 steels, respectively, indicating non-hardening-helium-embrittlement being more pronounced for ADS3 steel (due to larger helium amount produced).

Irradiation induced DBTT shift of EUROFER97 steel doped with 1120 wppm separated ^{10}B isotope could not be quantified due to large embrittlement found in the investigated temperature range. Such a large embrittlement, however, should not be only related to helium generation but also to the degraded impact properties in the unirradiated condition.

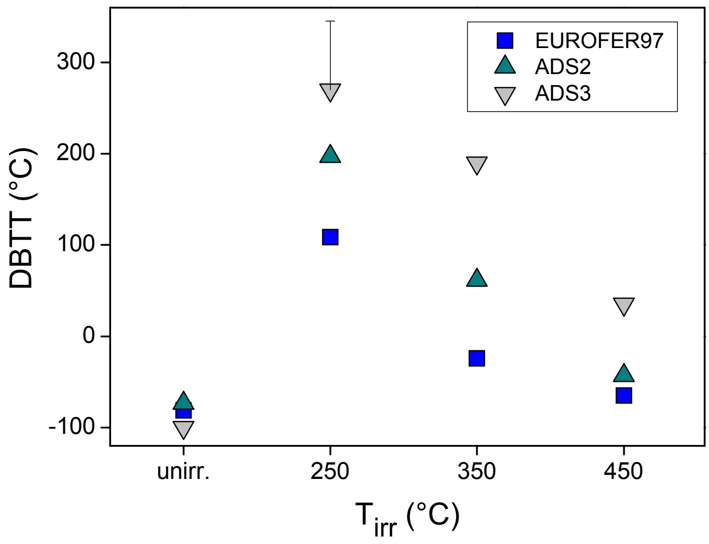

Fig. 3-12 Ductile-to-brittle transition temperature vs. irradiation temperature for EUROFER97 and ADS steels (irradiation programme SPICE: 16.3 dpa).

4 Summary

Collection and assessment of the available Charpy impact data on 7-10%-Cr-WVTa RAFM steels (EUROFER, F82H, OPTIFER-Ia etc.) has been performed. European reference RAFM steel EUROFER97 and international RAFM steels show superior irradiation performance compared to the modified commercial 10-11%-Cr-NiMoVNb steels, *e.g.* MANET I. Irradiation resistance of the RAFM steels for low irradiation temperatures $T_{irr} \leq 330$ °C, however, is not satisfactory, *i.e.* large, non-saturating low temperature embrittlement is seen up to 32 dpa. At 16.3 dpa/300 °C the irradiation resistance of EUROFER97 is comparable to that of Japanese reference steel F82H-mod (the DBTTs of RAFM steels lie between 100-110 °C in Fig. 3-7 for T_{irr} =300 °C), the latter, however, exhibits a broader ductile-to-brittle transition region. This may be partly explained by coarse and non-homogenous grain structure of F82H-mod. Heat treatment of the as-delivered EUROFER97 plate at higher austenization temperature (1040°C) leads to the reduction of the embrittlement for both 16.3 dpa/300 °C and 31.8 dpa/330 °C irradiation conditions, see Table 6-1. Close correlation between irradiation induced embrittlement and hardening at $T_{irr} \leq 350$ °C indicates displacement damage nature of low temperature embrittlement. Irradiation performance of EUROFER97 and international reference RAFM steels is satisfactory at $T_{irr} \geq 350$ °C up to 16.3 dpa.

Boron doped steels show progressive embrittlement and reduction of toughness with increasing He amount up to 5580 appm. He induced embrittlement is of hardening nature for low helium contents up to 84 appm. Larger helium concentrations lead to non-hardening embrittlement mechanisms beyond to the helium induced hardening. The extra embrittlement observed in boron doped steels, however, can not be solely related to produced He amounts.

The evolution of microstructure during steel irradiation is decisive for the steel irradiation performance.

5 References

[1] E. Gaganidze, Assessment of Fracture Mechanical Experiments on Irradiated EUROFER97 and F82H Specimens, FZKA 7310, 2007.

[2] M. Rieth, B. Dafferner, H. Ries, O. Romer, Bestrahlungsprogramm MANITU: Ergebnisse der Kerbschlagbiegeversuche mit den bis 0,8 dpa bestrahlten Werkstoffen der ersten Bestrahlungsphase, Forschungszentrum Karlsruhe, FZKA 5619, September 1995.

[3] M. Rieth, B. Dafferner, H. Ries, O. Romer, Bestrahlungsprogramm MANITU: Ergebnisse der Kerbschlagbiegeversuche mit den bis 0,2 dpa bestrahlten Werkstoffen, Forschungszentrum Karlsruhe, FZKA 5750, April 1997.

[4] H.-C. Schneider, M. Rieth, B. Dafferner, H. Ries, O. Romer, Bestrahlungsprogramm MANITU: Ergebnisse der Kerbschlagbiegeversuche mit den bis 0,8 dpa bestrahlten Werkstoffen der zweiten Bestrahlungsphase, Forschungszentrum Karlsruhe, FZKA 6519, September 2000.

[5] H.-C. Schneider, B. Dafferner, H. Ries, O. Romer, Bestrahlungsprogramm MANITU: Ergebnisse der Kerbschlagbiegeversuche mit den bis 2,4 dpa bestrahlten Werkstoffen, Forschungszentrum Karlsruhe, FZKA 6605, Mai 2001.

[6] H.-C. Schneider, B. Dafferner, J. Aktaa, Embrittlement behaviour of low-activation alloys with reduced boron content after neutron irradiation, J. Nucl. Mater. 321 (2003). 135-140.

[7] H.-C. Schneider, B. Dafferner, H. Ries, S. Lautensack, O. Romer, Bestrahlungsprogramm HFR Phase Ib: Ergebnisse der Kerbschlagbiegeversuche mit den bis 2,4 dpa bestrahlten Werkstoffen, Forschungszentrum Karlsruhe, FZKA 6976, April 2004.

[8] H.-C. Schneider, unpublished report, December 2002.

[9] E. Gaganidze, H.-C. Schneider, B. Dafferner, J. Aktaa, Journal of Nuclear Materials 355 (2006) 83-88.

[10] C. Petersen, A. Povstyanko, V. Prokhorov, A. Fedoseev, O. Makarov, B. Dafferner, Mechanical property degradation of ferritic/martensitic steels after the fast reactor irradiation 'ARBOR 1', Proceedings of ICFRM-12, December 4-9, 2005, Santa-Barbara, USA (in press).

[11] M. Rieth, M. Schirra, A. Falkenstein, P. Graf, S. Heger, H. Kempe, R. Lindau, H. Zimmermann: EUROFER97, Tensile, Charpy, Creep and Structural Tests, Forschungszentrum Karlsruhe, Wissenschaftliche Berichte FZKA 6911, October 2003.

[12] E. Lucon, Mechanical Properties of the European Reference RAFM Steel (EUROFER97) before and after Irradiation at 300 °C (0.3 - 2 dpa), SCK·CEN Report BLG-962, November 2003, Mol, Belgium.

[13] J. Rensman, NRG Irradiation Testing: Report on 300°C and 60°C Irradiated RAFM Steels, Petten 2005, 20023/05.68497/P.

[14] J. Rensman, J. Nucl. Mater. 307-311 (2002) 245-249.

[15] M. A. Sokolov et al., Proceedings of ICFRM-12, December 4-10, 2005, Santa-Barbara, USA (in press). M. A. Sokolov et al., Fusion Materials Semi-Annual Progress Reports, 2002, December 31, Volume 33, Page 59-65, DOE-ER-0313/33. R. L. Klueh, et al., J. Nucl. Mater. 307-311 (2002) 455-465. R.L. Klueh et al., J. Nucl. Mater. 283-287 (2000) 478-482.

[16] Yamamoto et al., J. Nucl. Mater. 356 (2006) 27-49.

[17] E. Gaganidze, B. Dafferner and J. Aktaa, Proceedings of 2006 MRS Fall Meeting, Nov 27 - Dec. 1, Boston, USA, MRS Symp. Proc. ; 981E, Electronic-Only Publication

6 Appendix – Impact Data on RAFM Steels

Material	Product form	Irradiation Campaign	Dose (dpa)	T_{irr} (°C)	DBTT (°C)	USE (J)	LTUS (°C)	Shift in DBTT (°C)
EUROFER97 ANL	25mm plate	unirr.	0	-	-81.3	9.84	-75	0
EUROFER97 ANL	25mm plate	SPICE	13.6	250	108.6	8.21	120	189.9
EUROFER97 ANL	25mm plate	SPICE	14.8	300	106.0	6.98	110	187.3
EUROFER97 ANL	25mm plate	SPICE	17.4	350	-24.2	8.86	-10	57.1
EUROFER97 ANL	25mm plate	SPICE	16.7	400	-62.0	9.13	-30	19.3
EUROFER97 ANL	25mm plate	SPICE	18.1	450	-65.0	9.33	-30	16.3
EUROFER97 ANL	25mm plate	WTZ 01/577	15.0	330	94.0	7.05	-	175.3
EUROFER97 ANL	25mm plate	ARBOR I	31.8	330	137.0	7.01	-	218.3
EUROFER97 WB	25mm plate	unirr.	0	-	-90.8	9.84	-75	0
EUROFER97 WB	25mm plate	SPICE	13.6	250	57.9	8.51	65	148.7
EUROFER97 WB	25mm plate	SPICE	17.4	350	-51.6	8.92	-15	39.2
EUROFER97 WB	25mm plate	SPICE	18.1	450	-64.4	9.26	-40	26.4
EUROFER97 WB	25mm plate	ARBOR I	31.8	330	107.0		-	197.8
OPTIFER Ia	23x23mm	unirr.	0	-	-80.0	10.1	-30	0
OPTIFER Ia	23x23mm	MANITU	0.2	300	-40.0	9.8	-20	40.0
OPTIFER Ia	23x23mm	MANITU	0.8	300	10.0	9.2	75	90.0
OPTIFER Ia	23x23mm	MANITU	2.4	300	30.0	8.7	60	110.0
OPTIFER Ia	23x23mm	unirr.	0	-	-81.3	10.6	-77	0
OPTIFER Ia	23x23mm	HFR Ia	2.4	300	-1.1	9.5	10	80.3
OPTIFER Ia	23x23mm	SPICE	14.8	300	103.9	8.14	115	185.2
OPTIFER Ia	23x23mm	SPICE	16.7	400	-45.0	9.42	23	36.3
OPTIFER IVc	40x40mm	unirr.	0	-	-105.0	8.75	-	0
OPTIFER IVc	40x40mm	ARBOR I	32.3	330	48.0	5.8	-	153.0
F82H	4mm plate	unirr.	0	-	-67.0	10.5	-65	0
F82H	4mm plate	MANITU	0.2	300	-50.0	10.5	-35.0	17.0
F82H	4mm plate	MANITU	0.8	300	-25.0	10.4	-20	42.0
F82H	4mm plate	MANITU	2.4	300	-15.0	9.8	0	52.0
F82H-mod	8mm plate	unirr.	0	-	-86.0	9.66	-77	0
F82H-mod	8mm plate	HFR Ib	2.4	300	17.0	7.91	50.0	103.0
F82H-mod	8mm plate	SPICE	14.8	300	110.0	6.74	150	196
F82H-mod	8mm plate	SPICE	16.7	400	-33.7	9.34	-10	52.3
F82H-mod	25mm plate	unirr.	0	-	-72.0	9.4	-	0
F82H-mod	25mm plate	ARBOR I	32.3	330	148.0	5	-	220
ORNL	4mm plate	unirr.	0	-	-85.0	9	-40	0
ORNL	4mm plate	MANITU	0.2	300	-60.0	8.6	-20	25.0
ORNL	4mm plate	MANITU	0.8	300	-45.0	8.4	-10	40.0
ORNL	4mm plate	MANITU	2.4	300	-40.0	8.2	-25	45.0
GA3X	4.5mm plate	SPICE	0	-	-57.5	9.35	-20.0	0.0
GA3X	4.5mm plate	SPICE	17.7	400	-65.0	9.05	-10.0	-7.4
GA3X	4.5mm plate	MANITU	0.8	300	-48.0	8.7	-48	14
GA3X	4.5mm plate	MANITU	0.8	400	-35.0	9.8	-20	27

Table 6-1 Impact properties of EUROFER97 and selected RAFM steels studied in [2]-[10]. KLST specimens are machined in LT orientation. EUROFER97 ANL: 980 °C/0.5 h + 760 °C/1.5 h; EUROFER97 WB: 1040 °C/0.5 h + 760 °C/1.5 h; F82H (MANITU: 1040 °C/0.5 h + 750 °C/2 h); F82H-mod (HFR Ib, SPICE: 950 °C/0.5 h + 750 °C/2 h, ARBOR 1: 1040 °C/38 min + 750 °C/2 h); DBTT: ductile-to-brittle-transition temperature; USE: upper shelf energy; LTUS: lowest temperature in the upper shelf.

Material	Product	Heat treatment	Orientation	Grain size (µm)	DBTT (°C)
EUROFER97	14 mm plate	980°C+760°C	transverse	16	-70
EUROFER97	14 mm plate	1050°C+750°C	transverse	26	-73
EUROFER97	14 mm plate	1075°C+750°C	longitudinal		-56
EUROFER97	14 mm plate	1075°C+750°C	transverse	45	-57
EUROFER97	∅100mm bar	980°C+740°C	longitudinal		-70
EUROFER97	∅100mm bar	980°C+740°C	transverse		-53

Table 6-2 Impact data on unirradiated EUROFER97 ISO-V Charpy specimens [11].

Irradiation group	Dose (dpa)	DBTT (°C)	Shift in DBTT (°C)
unirradiated	0	-57.3	0
1	0.34±0.066	-47.6	9.7
2	0.71±0.167	-37.5	19.8
3	1.55±0.313	1.5	58.8

Table 6-3 Analyses of impact data on unirradiated and 300 °C irradiated EUROFER97 ISO-V Charpy specimens with respect to the ductile-to-brittle transition temperature (DBTT). The irradiation induced shift in DBTT is also calculated [12].

Material	Product form	Irradiation Campaign	Dose (dpa)	T_{irr} (°C)	DBTT (°C)	USE (J)	Shift in DBTT (°C)
EUROFER97	14mm plate	unirr.	0	-	-90	9.3	0
EUROFER97	14mm plate	SUMO-04	1.95	300	-50	8.8	40
EUROFER97	8mm plate	unirr.	0	-	-86	9.0	0
EUROFER97	8mm plate	SUMO-04	2.23	300	-20	8.4	66
EUROFER97	8mm plate	SUMO-02	8.03	300	47	6.5	133
EUROFER97	100mm bar	unirr.	0	-	-86	9.0	0
EUROFER97	25mm plate	unirr.	0	-	-68	9.7	0
EUROFER97	25mm plate	SUMO-04	2.46	300	10	8.27	78
EUROFER97	25mm plate	SUMO-02	8.9	300	115	7.0	183
NRG 9Cr2WVTa-bis	plate	unirr.	0	-	-93	9.0	0
NRG 9Cr2WVTa-bis	plate	SUMO-02	8.7	300	30	7.04	123
NRG EUROFER	25mm plate	unirr.	0	-	-95	9.2	0
NRG EUROFER	25mm plate	SUMO-02	6.99	300	10	7.58	105
NRG EUROFER	25mm plate	SIWAS-09	2.33	60	-15	9.29	80
F82H	15mm plate	unirr.	0	-	-82	9.0	0
F82H	15mm plate	SUMO-04	2.34	300	60	8.0	142
F82H	15mm plate	CHARIOT-02	2.64	300	60	8.7	142
F82H	15mm plate	SIWAS-06	2.07	60	35	8.5	117
F82H	25mm plate	unirr.	0	-	-89	9.5	0
F82H	25mm plate	CHARIOT-04	2.64	300	11	9.0	100
F82H	25mm plate	SUMO-02	8.5	300	98	6.0	187
F82H opt. HT.	25mm plate	unirr.	0	-	-109	9.9	0
F82H opt. HT.	25mm plate	SUMO-04	2.34	300	0	9.9	109
F82H opt. HT.	25mm plate	CHARIOT-04	2.5	300	10	9.0	119

Table 6-4 Analyses of KLST impact test on the unirradiated and irradiated EUROFER97 and other reference RAFM steels studied in [13],[14]. The 25 mm plates of F82H have been tested in the as-received and in the optimum heat-treated (opt. HT.) conditions.

Material	Orientation	Specimen (mm^3)	Dose (dpa)	T_{irr} (°C)	USE (J)	DBTT (°C)	Shift in DBTT (°C)
F82H-IEA		3.3x3.3x25.4	0	-	11.8	-85	0
F82H-IEA		3.3x3.3x25.4	5	500	11.5	-60	25
F82H-IEA		3.3x3.3x25.4	20	500	12.2	-50	35
F82H-IEA		3.3x3.3x25.4	5	300	10.3	25	110
F82H-IEA		3.3x3.3x25.4	20	380	9.2	50	135
F82H-HAT		3.3x3.3x25.4	0	-	14.8	-105	0
F82H-HAT		3.3x3.3x25.4	5	500	12.1	-95	10
F82H-HAT		3.3x3.3x25.4	5	300	11.9	5	110
F82H-HAT		3.3x3.3x25.4	20	380	8.5	32	137
F82H-Std	LT	3.3x3.3x25.4	0	-	12.3	-103	0
F82H-Std	LT	3.3x3.3x25.4	10	300	7.9	56	159
F82H-Std	LT	3.3x3.3x25.4	12	400	9.7	14	117
F82H-mod	LT	3.3x3.3x25.4	0	-	10.8	-82	0
F82H-mod	LT	3.3x3.3x25.4	9	300	8.3	70	152
F82H-mod	LT	3.3x3.3x25.4	11	400	8.3	64	146
9Cr-2WVTa	LT	3.3x3.3x25.4	0	-	10.8	-94	0
9Cr-2WVTa	LT	3.3x3.3x25.4	11	400	6.5	-15	79

Table 6-5 Impact properties on F82H in the as received (IEA 1040 °C/40 min+ 750 °C/1 h) and heat treated (HT: 920 °C/1 h + 750 °C/ 1h) conditions [15]. The 1/3 size Charpy V specimens (3.3x3.3x25.4 mm^3) have been tested in the unirradiated and irradiated conditions. The DBTT was obtained at half the upper-shelf value. For F82H-Std and F82H-mod steels, austenitization was at 1040 °C followed by tempering 1 h at 750 °C. The 9Cr-2WVTa was austenitized at 1050 °C followed by tempering 1 h at 750 °C.

Heat	B-content	He-generation
EUROFER97	10 wppm nat. B	10 appm
ADS2	82 wppm nat. B	84 appm
ADS3	83 wppm ^{10}B	432 appm
ADS4	1120 wppm ^{10}B	5580 appm

Table 6-6 B content and irradiation induced He generation in EUROFER97 and boron doped steels. Nat. B = ^{11}B (80%) + ^{10}B (20%).