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Assessment of Fracture Mechanical Experiments on Irradiated EUROFER97 and F82H Specimens

E. Gaganidze

Institut für Materialforschung
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E. Gaganidze

Institut für Materialforschung

Association Forschungszentrum Karlsruhe / EURATOM

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Forschungszentrum Karlsruhe GmbH
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Auswertung von bruchmechanischen Untersuchungen an bestrahlten EUROFER97 und F82H Proben

Zusammenfassung

Der vorliegende Bericht fasst die Auswertung bruchmechanischer Untersuchungen zusammen, die an unterschiedlichen weltweiten Institutionen an unterschiedlichsten Proben aus bestrahltem EUROFER97 sowie weiteren bestrahlten niedrigaktivierbaren Stählen (F82H, etc.) durchgeführt wurden. Die Arbeit wurde im Rahmen des europäischen Abkommens zur Entwicklung der Fusionsforschung (EFDA), Subtask TW5-TTMS-001-D14, vorgenommen. Die Auswertung der Untersuchungen der bestrahlten Stähle wurde in erster Linie anhand von Ergebnissen aus quasistatischen 3-Punkt-Biege-Versuchen an angerissenen Charpy V Proben (Pre-Cracked Charpy V: PCCV) und Zugversuchen an Kompaktproben (CT) vorgenommen. Dabei wurde unter anderem auch eine eventuelle Abhängigkeit der Ergebnisse vom Probentyp und der Probengröße betrachtet. Zusätzlich zu den bruchmechanischen Tests wurden aus Vergleichsgründen hinsichtlich der bestrahlungsinduzierten Verschiebung der Übergangstemperaturen auch Auswertungen von Versuchen an Charpy Proben durchgeführt.

Die Bestimmung der bruchmechanischen Übergangstemperaturen erfolgte durch die Anwendung des *Master Curve* Ansatzes an den normierten Werten der Bruchzähigkeit. Die Auswertung der bruchmechanischen Untersuchungen zeigt eine zunehmende Versprödung von EUROFER97 mit zunehmender Bestrahlungsdosis (bis 9 dpa). Folglich verschiebt sich auch die Übergangstemperatur hin zu höheren Temperaturen. Ein Sättigungsverlauf kann zwar nicht beobachtet werden, die Ergebnisse aus den bruchmechanischen Experimenten sind allerdings konsistent. Ein Einfluss des Probentyps (PCCV, CT) oder der Probengröße (5mm, 10mm) ist nicht erkennbar. Im Gegensatz dazu zeigen Kerbschlagbiegeversuche an KLST und ISO-V Proben deutlich geringere Verschiebungen der jeweiligen Übergangstemperaturen. Aber auch die Ergebnisse aus diesen Untersuchungen sind hinsichtlich der Materialversprödung in sich konsistent. Die bestrahlten F82H Stähle zeigen eine starke Streuung von bruchmechanischen Übergangstemperaturen. Dieses Verhalten verhinderte eine eindeutige quantitative Analyse des Versprödungsverlaufes mit der Bestrahlungsdosis.

Abstract

This report summarizes assessment of Fracture Mechanical (FM) experiments carried out by different worldwide institutions on irradiated EUROFER97 and other RAFM steels (F82H, etc.). The assessment was carried out under the contract of the European Fusion Development Agreement (EFDA), subtask TW5-TTMS-001-D14. The results on irradiated steels obtained in different kinds of FM tests, *i.e*. in quasi-static *three-point-bend* tests on Pre-Cracked Charpy V (PCCV) specimens and in tension tests on compact-tension (CT) specimens have been assessed. Among other things the effects of specimen type and specimen size on the fracture mechanical results have been investigated. A comparison of fracture mechanical and Charpy tests with respect to the DBTT and irradiation induced shift in the DBTT was done in addition.

The Master Curve methodology has been applied to normalized fracture toughness data for determination of fracture toughness transition temperature. Analysis of FM experiments shows a progressive embrittlement of EUROFER97 with increasing irradiation dose (up to 9 dpa). The transition temperature shifts to higher temperatures with irradiation with no sign of saturation. The transition temperature appears to be independent of specimen type (PCCV, CT) and specimen thickness (5mm, 10mm). The irradiation induced shifts in transition temperatures obtained in FM experiments on EUROFER97 are consistently and significantly larger than the corresponding shifts quantified by impact testing on KLST and ISO-V specimens. Though, the results of impact testing with respect of material embrittlement are consistent. Irradiated F82H steels exhibit large scattering of fracture toughness transition temperature. This behaviour prevented unambiguous quantitative analysis of the embrittlement trend.

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1 Objective

Various fracture mechanical (FM) experiments have been performed on irradiated EUROFER97 and other reduced activation ferritic-martensitic (RAFM) steels (F82H, OPTIFER). As different specimen types and testing methods were used for specimens irradiated at different conditions, an overview with directly comparable parameters is missing. The assessment performed in the current work aims normalization of available fracture toughness data for direct comparison to study the specimen geometry and specimen size effects. Charpy impact testing on KLST type specimens is a widely used testing method for characterization of the material irradiation resistance. This led to the development of a large data base on the ductile-to-brittle transition temperature (DBTT) in a wide irradiation dose range. The study of correlation of fracture mechanical and impact testing results is an important step for anticipation of FM results for not (or not yet) available irradiation conditions.

2 Data Collection

Data has been collected from associations having performed irradiated FM and 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/0.5h 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, nominal composition 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 optimum heat-treated conditions.

Other RAFM Steels

An experimental heat of ORNL 9Cr-2WVTa (nominal composition Fe-9Cr-2W-0.25V-0.07Ta-0.01C) 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.

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.

2.2 Fracture Mechanical and 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 [1]. Specimens have been fabricated from the ∅=100 mm bars.

Fracture Mechanical *three-point-bend* tests were performed, in the unirradiated and irradiated conditions, on pre-cracked Charpy V (PCCV) plain-sided (non side-grooved) specimens (10x10x55 mm^3) for determination of fracture toughness K_{Jc} (an elastic-plastic equivalent stress intensity factor derived from the J integral at the point of onset of cleavage fracture, J_c). Tests have been carried out at several temperatures within the transition region in order to obtain the reference temperature T_o in accordance with the ASTM E1921-02 standard (multi-temperature approach). The specimens have been fatigue pre-cracked to relative crack lengths of $a_0/W \approx 0.5$, with a_0 being the initial crack length and W the specimen width. Taking into account individual fluence values, the following sub-groups was considered for analyses: Sub-group 1 (0.33±0.034 dpa), Sub-group 2 (0.74±0.191 dpa) and Sub-group 3 (1.62±0.333 dpa). Fig. 6-1 shows the experimental data normalised to 1T CT thickness (B_{1T}=25.4 mm) together with corresponding Master Curves. A progressive material embrittlement with increasing irradiation dose is seen as can also be inferred from Table 6-1.

Impact tests were done on standard Charpy V-notch specimens (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: 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 impact test results on the unirradiated and irradiated specimens are shown in Fig. 6-2. A progressive material embrittlement with increasing irradiation dose is observed as can be inferred from Table 6-2.

2.3 Fracture Mechanical and Impact Testing at NRG

Fracture toughness and impact properties on unirradiated and 2.13, 2.5, 10 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-07, SIWAS-09, SINAS-80/6, SINAS-80/7) [2]. Irradiation was performed at 300 °C and 60 °C.

The fracture toughness experiments have been performed on reduced size compact tension (CT) specimens of dimensions of 5x27x29 mm^3 (CT5) and 10x27x29 mm^3 (CT10). The specimens have been first fatigue pre-cracked to relative crack lengths of a_0/W = 0.5. Prior to testing selected specimens have been side grooved, in order to improve a constraint state. The ASTM standard 1921-03 was followed with regard to the calculation of fracture toughness (K_{Jc}). The fracture mechanical and impact EUROFER97 specimens have been machined from 8, 14 and 25 mm plates. Some KLST specimens have been machined from 100 mm round bar as well. The fracture mechanical and impact F82H-mod specimens have been machined from 15 and 25 mm plates.

The fracture toughness results on unirradiated and 300 °C irradiated EUROFER97 and NRG Eurofer lab heat specimens are shown in Fig. 6-3. The 8 mm and 14 mm plate results were undistinguishable. Relatively large data scattering can be seen already in the unirradiated condition, partly explained by statistical nature of fracture. Different fracture toughness results observed for different base materials (different plate thicknesses) indicate toughness dependence on steel/specimen fabrication details. The 25 mm plate specimens show low fracture toughness at a given test temperature. A progressive material embrittlement with the irradiation dose is observed in a good qualitative agreement with [1]. More pronounced data scattering compared to the unirradiated condition can partly be due to the variation in the achieved doses, since no saturation of irradiation hardening is expected at this temperature.

In [3] Rensman reported fracture toughness data on unirradiated and 300 °C irradiated F82H-mod, obtained within the SINEXT-06 irradiation campaign. The results reveal large irradiation induced material embrittlement as seen in Fig. 6-4.

The fracture toughness results on 60 °C irradiated EUROFER97, F82H, NRG Eurofer and NRG 9Cr2WVTa lab heat are shown in Fig. 6-5. Somewhat lower scatter of irradiated data is observed compared to the 300 °C irradiation, see Fig. 6-4.

Some J-R tests have been performed as well, in accordance with the ESIS P-92 procedure for upper shelf fracture toughness. Reduction of fracture toughness as well as the tearing resistance (dJ/da) was observed for 2.14dpa/60°C irradiated EUROFER97 25 mm plate.

The results of KLST impact tests on unirradiated and irradiated EUROFER97 and other RAFM steels are summarised in Table 6-3.

2.4 Fracture Mechanical and Impact Testing at ORNL

ORNL intensively studied fracture toughness and impact properties of several RAFM steels [4]. F82H, F82H with 2% Ni, 9Cr-2WVTa steels have been irradiated in the ORNL High Flux Isotope Reactor (HFIR). The fracture mechanical experiments have been performed on miniaturised disc-shaped compact tension (DCT) specimens (12.5 mm in diameter with thickness of 4.6 mm) and on pre-cracked 1/3-size Charpy V (PCCV 3.3x3.3x25.4 mm^3) specimens. The fracture toughness tests were conducted in accordance with the ASTM

E1921-02 standard for determination of reference temperature T_o. Before irradiation the (DC(T)) specimens were fatigue pre-cracked to a relative length of $a_0/W \approx 0.5$, and then side-grooved by 20 % of their thickness (10% from each side). Impact tests were performed on miniaturised, 1/3-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 6-4 shows Master Curve analyses of fracture toughness results on the F82H IEA heats in the as-received and heat treated (HT) conditions. The specimens have been machined in TL and LT orientations and subsequently irradiated up to 5 dpa at target temperatures of 300 °C and 500 °C. Additionally, J-R testing of RAFM steels irradiated up to 3.8 dpa between 240 and 340 °C (F82H HT, 9C2WVTa) revealed reduction of tearing resistance and J_c compared to the unirradiated state.

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 have been machined in the L-T orientation and subsequently irradiated at 300, 380 and 500 °C target temperatures. An additional heat treatment 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.

2.5 Fracture Mechanical and Impact Testing at FZK

Quasi-static fracture toughness tests on EUROFER97 have been performed only in the unirradiated states using three-point-bend specimens of different geometries (3x4x27 mm^3, 3x6x30 mm^3, 9x18x92mm^3 [5], [6].

Impact properties of EUROFER97 and other RAFM (F82H, F82H-mod, OPTIFER series) steels have been intensively studied at FZK in different irradiation programmes (MANITU, HFR Phase Ia, Ib, IIa, IIb (SPICE), ARBOR I, II) up to 32.8 dpa [7],[8],[9],[10],[11],[12],[13],[14]. Reduced sized Charpy V specimens of KLST type 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 6-6 summarises impact properties of KLST specimens. Additionally, a thorough investigation of the reference unirradiated state of EUROFER97 was performed with standard ISO-V Charpy specimens [15].

3 Data Assessment

3.1 Master Curve Methodology

For application of Master Curve analysis the fracture toughness data is thickness adjusted to the reference 1T CT specimen thickness (B_{1T} =25.4 mm) according to statistical weakest link model by the relationship

$$K_{Jc(1T)} = K_{\min} + \left[K_{Jc(x)} - K_{\min}\right]\left(\frac{B_x}{B_{1T}}\right)^{1/4} \tag{1}$$

Here, $K_{Jc(x)}$ is the fracture toughness as measured on a specimen of a thickness B_x, $K_{\min}$ is the lower bound fracture toughness fixed at 20 MPa·m$^{0.5}$ in ASTM E1921-05 [16] and $K_{Jc(1T)}$ is the normalized fracture toughness.

For *single temperature* data the *Weibull* fitting parameter K_o (located at the 63.2 % cumulative failure probability) level is calculated with all normalized $K_{Jc(i)}$ and dummy values by

$$K_o = \left[\sum_{i=1}^{N} \frac{(K_{Jc(i)} - K_{\min})^4}{r}\right] + K_{\min} \tag{2}$$

where r is the number of valid data and N is the number of all data.

The maximum K_{Jc} capacity of a specimen

$$K_{Jc(limit)} = \sqrt{\frac{Eb_o\sigma_{ys}}{30(1-\nu^2)}} \tag{3}$$

has to be used as dummy value in the case of not sufficient specimen remaining ligament b_o. Here E is the Young's modulus for plane stress, σ_{ys} is the material yield strength at the test temperature and ν (≈0.3) is the Poisson's ratio.

The fitting parameter K_o is converted to $K_{Jc(med)}$ value (corresponds to 50% cumulative probability for fracture) by

$$K_{Jc(med)} = K_{\min} + \left(K_o - K_{\min}\right)\left[\ln(2)\right]^{1/4} \tag{4}$$

The reference temperature T_o is calculated using the Master Curve (MC) methodology

$$T_o = T - \left(\frac{1}{0.019}\right)\ln\left[\frac{K_{Jc(med)} - 30}{70}\right] \tag{5}$$

For *multi-temperature* data the reference temperature T_o is calculated with all normalized $K_{Jc(i)}$ and dummy values from the following equality:

$$\sum_{i=1}^{N} \delta_i \frac{\exp\left[0.019(T_i - T_o)\right]}{11 + 77\exp\left[0.019(T_i - T_o)\right]} - \sum_{i=1}^{N} \frac{\left(K_{Jc(i)} - 20\right)^4 \exp\left[0.019(T_i - T_o)\right]}{\left\{11 + 77\exp\left[0.019(T_i - T_o)\right]\right\}^5} = 0 \tag{6}$$

where, T_i is the test temperature corresponding to $K_{Jc(i)}$, δ_i is 1.0 if the datum is valid or zero if the datum is a dummy substitutive value.

3.2 EUROFER97

Fig. 3-1 shows uncensored fracture toughness data on EUROFER 97 for unirradiated and irradiated conditions [1],[2]. For comparison of different specimen results, the fracture toughness data were adjusted to reference 1T CT thickness according to Eq. (1). In the unirradiated condition NRG data on the 8 mm and 14 mm plates fall in the same scattering band for both specimen thicknesses. The 25 mm plate specimens, however, show lower toughness at a given temperature, or equivalently, a higher transition temperature. The unirradiated SCK data agree well with the unirradiated NRG data, remaining in the same scattering band. The NRG data show progressive embrittlement with irradiation dose from 2.13 to 8.81 dpa. Data scattering is increased for irradiated condition partly due to different damage doses received by individual specimens. The SCK data for irradiated doses between 0.33 and 1.62 dpa confirm qualitatively the irradiation induced embrittlement trend observed for the irradiated NRG data.

In order to quantify the effects of specimen geometry and/or specimen size on the fracture toughness transition temperature, ASTM E1921-05 procedure has been applied to 1T CT thickness adjusted fracture mechanical data [2] which were grouped with respect to material, product form, irradiation condition, specimen type (i.e. PCCV or CT) and thickness (5 mm or 10 mm), as shown in Table 6-7. For determination of reference temperature all invalid K_{Jc} data were censored and replaced by the dummy $K_{Jc(limit)}$ values determined by Eq. (3). The censoring of the data with respect to the maximum crack growth criteria of [16] has not been performed. The results of ASTM E1921-05 analysis for determination of T_o are summarized in Table 6-7. In the unirradiated condition and for 25 mm EUROFER97 plate T_o determined on 5 and 10 mm CT specimens are nearly identical as inferred from Table 6-7. Furthermore, for the same 25 mm EUROFER97 plate and for 2.5dpa@300 °C irradiation slightly different T_o values of 38.4 and 26.2 °C are obtained on 5 and 10 mm CT specimens, respectively. The

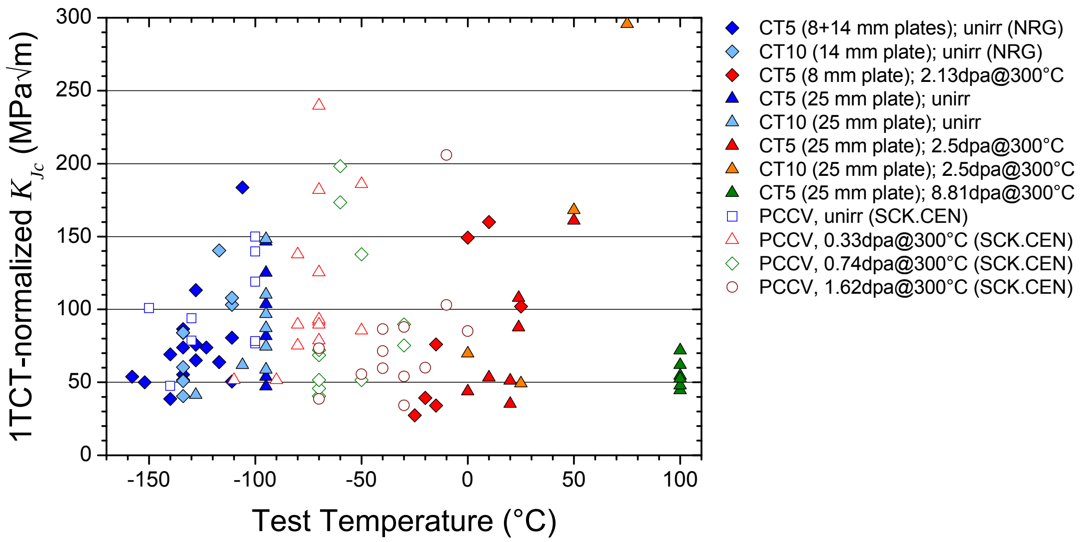

Fig. 3-1 1T CT thickness adjusted fracture toughness data on EUROFER97 from [1],[2]. Specimen type (base material), irradiation condition and data sources are indicated in the legend.

availability of *three* of *four* valid K_{Jc} results only, however, makes T_o for 10 mm thick CT specimens *invalid* as a minimum of six valid K_{Jc} data are required according to [16].

Fig. 3-2 shows the fracture toughness transition temperature T_o vs. irradiation dose for EUROFER97 (results from Table 6-1 and Table 6-7). A progressive material embrittlement with irradiation dose is observed with no saturation up to 8.8 dpa. The transition temperature appears to be independent of specimen type (PCCV, CT) or specimen thickness (5mm, 10mm). The dose dependence of transition temperature is well described with a function of type

$$T_o = A_1 + A_2\left[1 - \exp\left(-\frac{dose}{\tau}\right)\right] \tag{7}$$

The best fit was obtained with $A_1 = -107.4$ °C, $A_2 = 297.8$ °C, $\tau = 4.23$ dpa.

In order to analyse the effect of available product form on the steel irradiation resistance the fracture toughness data have been grouped with respect to material and product form as well as their irradiation condition. Additionally, the results on 8 and 14 mm plate were grouped together since they were undistinguishable [2]. The results of application of ASTM E1921-05 procedure to the 1T CT normalized data are presented in Table 6-8 and in Fig. 3-3. The irradiation induced shift in T_0 in Fig. 3-3 is best described with a function of type

$$\Delta T_o = A\left[1 - \exp\left(-\frac{dose}{\tau}\right)\right] \tag{8}$$

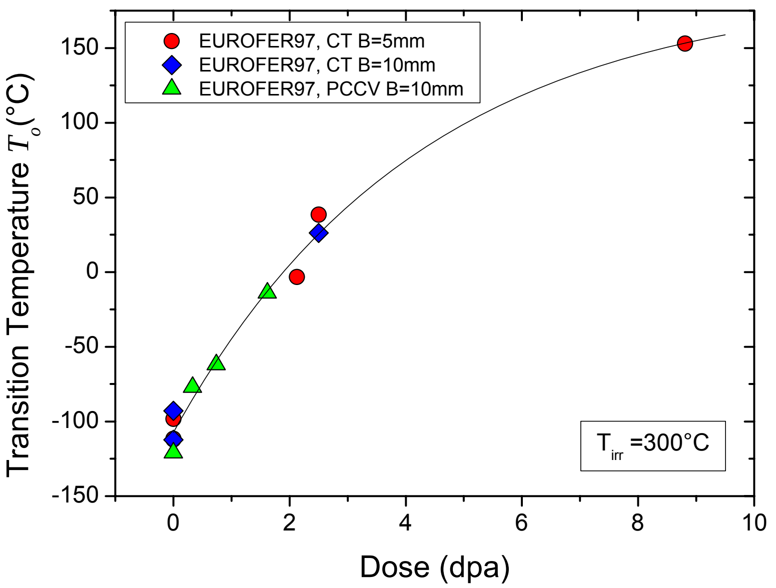

Fig. 3-2 Fracture toughness transition temperature vs. irradiation dose on EUROFER97 (see Table 6-1 and Table 6-7). Compact Tension (CT) specimens were machined from 8, 14 and 25 mm plates. Pre-Cracked Charpy V (PCCV) specimens were machined from ∅=100mm bars. The specimen thicknesses are indicated in legend. The line is a least square fit to data with Eq. (7) with $A_1 = -107.4$ °C, $A_2 = 297.8$ °C, $\tau = 4.23$ dpa.

The best fit was obtained with fitting parameters $A = 268.9$ °C, $\tau = 3.58$ dpa.

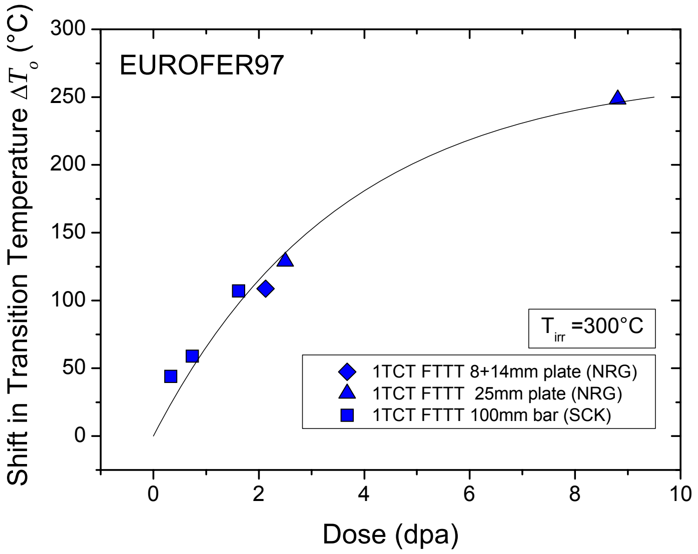

Fig. 3-3 Shift in fracture toughness transition temperature vs. irradiation dose on EUROFER97 (see Table 6-8). The fracture toughness data are grouped according to product form and irradiation condition. The line is a least square fit to data with Eq.(8). A=268.9 °C, $\tau = 3.58$ dpa.

3.3 F82H

ASTM E1921-05 procedure has been applied to 1T CT thickness adjusted fracture toughness data on F82H-mod from [2],[3]. Table 6-9 summarizes the results on 15 and 25 mm plates. Fig. 3-4 shows the fracture toughness transition temperature T_0 from Table 6-9 together with the results on unirradiated and irradiated (60°C≤T_{irr}≤300°C) F82H-IEA from Table 6-4. Large differences in T_o are observed already in the unirradiated condition. Experiments in TL orientation show larger T_o compared to LT orientation. Furthermore, for 5dpa@300°C irradiated steels, T_o determined in *three-point-bend* tests with 1/3-size V-notch specimens (ORNL) is much smaller than the corresponding value determined with CT specimens (NRG). The absence of systematic studies, however, prevents generalization of the observed disagreement. Comparatively, fracture mechanical experiments with CT and full size PCCV specimens on unirradiated and irradiated EUROFER97 yielded comparable T_o values, as shown in Fig. 3-2.

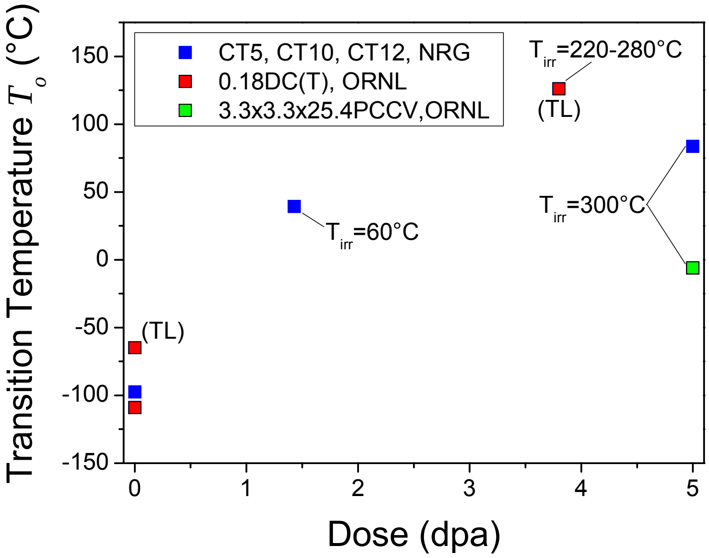

Fig. 3-4 Fracture toughness transition temperature vs. irradiation dose on F82H (see Table 6-4 and Table 6-9). T_{irr}= 60-300°C. Specimen type is indicated in figure legend. If not otherwise indicated the specimens are machined in LT orientation.

Fig. 3-5 shows irradiation induced shift in transition temperature vs. irradiation dose for F82H, see also Table 6-4 and Table 6-9. No reference temperature in the unirradiated state was available for 1/3-size PCCV specimens in [4]. Because of this T_o determined with unirradiated DC(T) specimens was used as reference for coarse estimation of irradiation

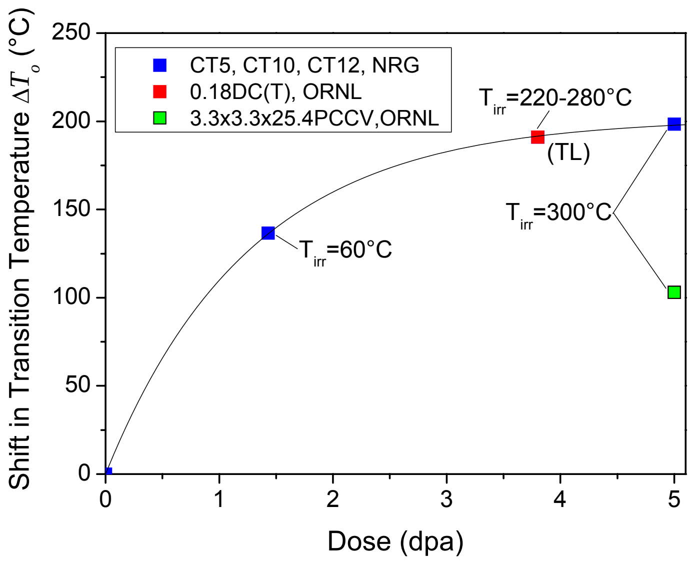

Fig. 3-5 Shift in fracture toughness transition temperature vs. irradiation dose for F82H (see Table 6-4 and Table 6-9). T_{irr}=60-300°C. If not otherwise indicated the specimens were machined in LT orientation. The line is a least square fit with Eq. (8) to the data points obtained with CT and DC(T) specimens, see text. $A = 201.7$°C, $\tau = 1.27$dpa.

induced shift for 5dpa@300°C irradiated PCCV specimens in Fig. 3-5. The shift in T_o estimated this way is much smaller than the corresponding shift quantified with CT specimens for 5dpa@300°C irradiation. By neglecting the ΔT_o data point for a 1/3-size PCCV specimen, a progressive material embrittlement with the irradiation dose is observed for F82H steel. The line in Fig. 3-5 is a least square fit with Eq. (8) to the data obtained with CT and DC(T) specimens, only. The best fit was obtained with the fitting parameters A=201.7 °C, τ=1.27 dpa.

3.4 Comparison of Fracture Toughness Transition Temperatures between EUROFER97 and other RAFM Steels

F82H

Fig. 3-6 shows the fracture toughness transition temperature for EUROFER97 and F82H steels vs. irradiation dose. Due to large scattering in T_o for F82H no unambiguous comparison can be done between the irradiation dose dependences of T_o of the two steels.

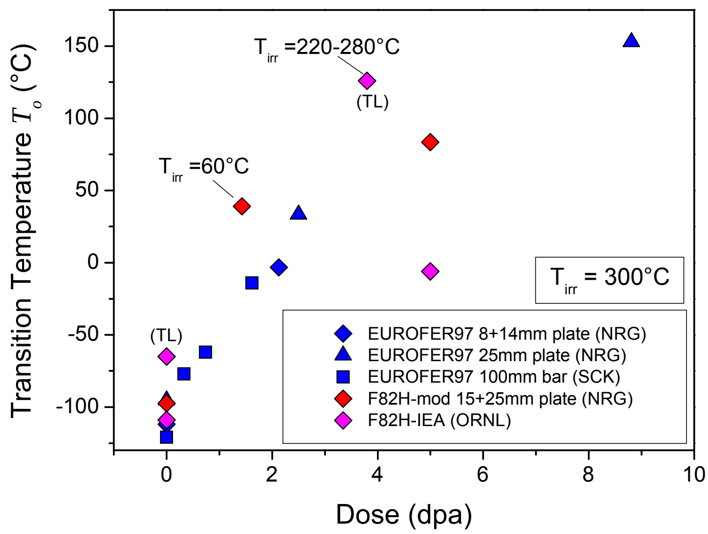

Fig. 3-6 Fracture toughness transition temperature for EUROFER97 and F82H steels vs. irradiation dose. If not otherwise indicated the specimens were machined in LT orientation.

NRG EUROFER Lab Heat

Fig. 3-7 shows fracture toughness transition temperature for EUROFER97 and NRG EUROFER Lab Heat vs. irradiation dose, see also Table 6-7. The irradiation is performed at 300°C. At irradiation dose of 2.12 dpa NRG EUROFER Lab Heat shows somewhat lower transition temperature in comparison with EUROFER97. At irradiation dose of 7.42 dpa, however, the transition temperature of NRG EUROFER Lab Heat lies much below the line describing embrittlement of EUROFER97 (the line in Fig. 3-7 is a least square fit to EUROFER97 data with

Eq. (7)). This observation is in a qualitative agreement with impact test results in [2], where EUROFER97 25mm plate showed lower impact toughness than NRG EUROFER Lab Heat.

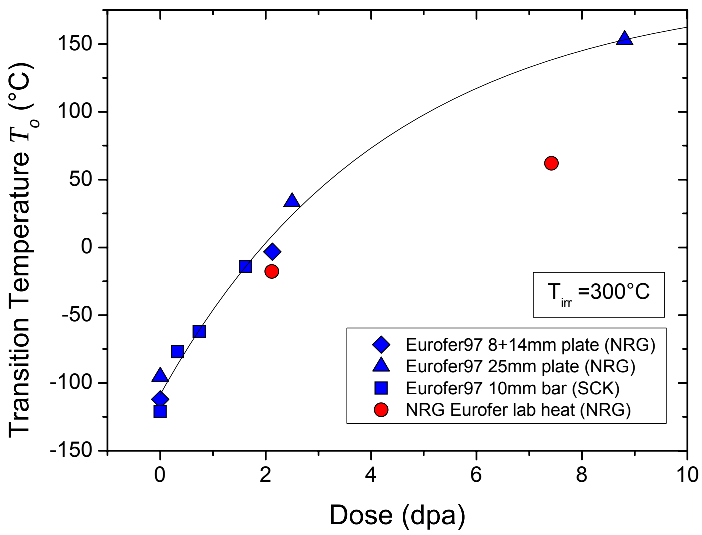

Fig. 3-7 Fracture toughness transition temperature for EUROFER97 and NRG EUROFER Lab Heat vs. irradiation dose. The line is a least square fit to EUROFER97 data with Eq. (7). $A_1 = -108.53$ °C, $A_2 = 300.27$ °C, $\tau = 4.3$ dpa.

3.5 Comparison of Shifts in Transition Temperatures from Fracture Mechanical and Impact Tests

Fig. 3-8 shows irradiation induced shifts in the transition temperatures of EUROFER97 quantified in FM and impact experiments. The irradiation induced shifts in transition temperatures obtained in FM experiments up to 8.8 dpa are consistently and significantly larger than the corresponding shifts quantified by impact testing on KLST specimens. Consequently, the embrittlement behaviour quantified by impact testing on KLST specimens is non-conservative and has to be handled with precaution. Remarkably, the same conclusion was drawn in [1] by comparative FM (standard PCCV) and impact (standard ISO-V Charpy) studies on EUROFER97 up to 1.6 dpa. Those impact data on standard ISO-V Charpy specimens are also included in Fig. 3-8 for comparison. The results of impact testing with respect of material embrittlement are consistent for KLST and ISO-V specimens. Large data scattering observed for KLST specimens at low and intermediate irradiation doses in Fig. 3-8 prevents a quantitative assessment of embrittlement trend quantified by impact testing. The dashed line is a least square fit with Eq. (8) to the impact DBTT data (ISO-V, KLST) that are weighted with irradiation dose (weighting factors $\propto$ Dose). The best fit is obtained with A=217.23 °C and τ=7.15 dpa. The solid line is the least square fit to the FTTT data with Eq.(8) as described in Fig. 3-3. The two lines in Fig. 3-8 may be used for the first estimation of the relationship between the FTTT and DBTT shifts for EUROFER97. The following relationship can be derived between irradiation induced shifts in T_o and in DBTT

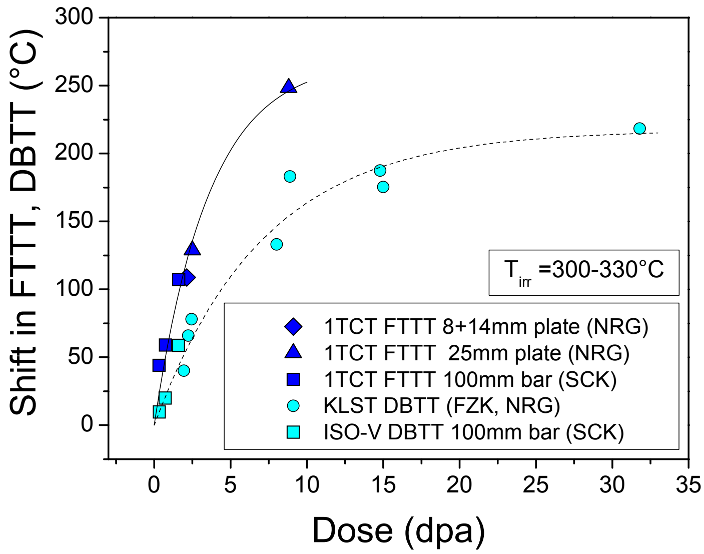

Fig. 3-8 Irradiation induced shift in Fracture Toughness Transition Temperature (FTTT) and KLST and ISO-V Ductile-to-Brittle Transition Temperature (DBTT) for EUROFER97 vs. irradiation dose. The solid line is a least square fit to FTTT data with Eq. (8). A=268.9 °C, τ=3.58 dpa. The dashed line is a least square fit to DBTT data with Eq. (8) using weighting of the data with respect to irradiation dose, see text. A=217.23 °C and τ=7.15 dpa.

$$\Delta T_o = A_{FTTT}\left\{1-\left(1-\frac{\Delta DBTT}{A_{DBTT}}\right)^{\frac{\tau_{DBTT}}{\tau_{FTTT}}}\right\} \quad (9)$$

where, parameters A and τ as defined in Eq. (8) now have indices corresponding to FTTT and DBTT shift results. By definition of the parameters in Eq. (8), Eq. (9) can only be used for $\Delta DBTT \leq A_{DBTT}$ (or for $\Delta T_o \leq A_{FTTT}$ if the inverse formula is used). Taking into account the determined A and τ values for EUROFER 97 (i.e. those from Fig. 3-8), one obtains

$$\Delta T_o = 268.9\left\{1-\left(1-\frac{\Delta DBTT}{217.3}\right)^{2.0}\right\} \quad (10).$$

Large scattering of FM and impact data on F82H, see Table 6-3, Table 6-5 and Table 6-6 prevented determination of unambiguous correlation between the irradiation induced transition temperature shifts quantified by FM and impact experiments.

4 Summary

Collection and assessment of the available fracture toughness and Charpy impact data on RAFM steels (EUROFER, F82H) has been performed. The effects of specimen type, specimen size, test method, etc. have been investigated. MC methodology has been applied to normalized fracture toughness data for determination of FTTT. FTTT appeared to be independent of specimen type (CT, PCCV) and size (B=5-10 mm). Progressive material embrittlement observed for EUROFER97 indicate no saturation for the achieved damage doses. The irradiation induced shifts in transition temperatures obtained in FM experiments on EUROFER97 are consistently and significantly larger than the corresponding shifts quantified by impact testing on KLST and ISO-V specimens. Though, the results of impact testing with respect of material embrittlement are consistent. Irradiated F82H steels exhibit large scattering of fracture toughness transition temperature. This behaviour prevented unambiguous quantitative analysis of the embrittlement trend.

5 References

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6 Appendix

6.1 Collection of Fracture Mechanical and Impact Data on RAFM Steels

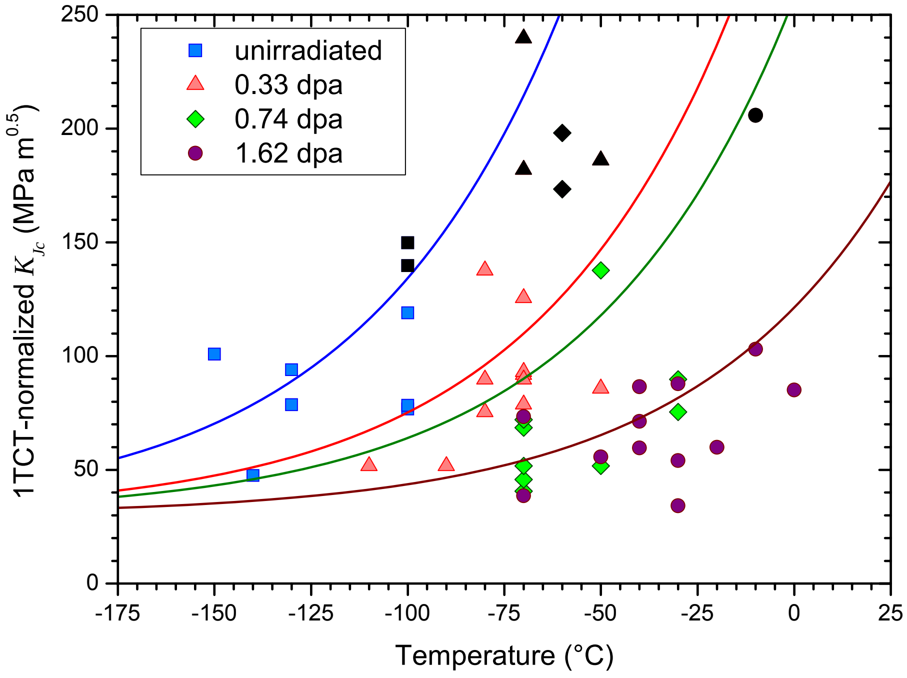

Fig. 6-1 1T CT normalized fracture toughness data on unirradiated and 300 °C irradiated EUROFER97 along with corresponding Master Curves [1]. Black solid points correspond to invalid data according to ASTM E1921.

Irradiation group	Dose (dpa)	N	r	K_o (MPa√m)	K_{med} (MPa√m)	T_o (°C)	Shift in T_o (°C)
unirradiated	0	9	7	119.8	111.1	-121.1	0
1	0.33 ± 0.034	15	12	118.1	109.5	-76.6	44.5
2	0.74 ± 0.191	11	9	118.8	110.1	-62.1	59.0
3	1.62 ± 0.333	13	12	88.1	82.1	-14.5	106.6

Table 6-1 Results of Master Curve analyses of fracture toughness results on unirradiated and irradiated EUROFER97 pre-cracked Charpy V (PCCV) specimens (10x10x55 mm^3) [1]. N is number of tests performed, r number of valid tests, K_o Weibull fitting parameter (corresponding to 63% probability for specimen failure by cleavage, K_{med} median fracture toughness corresponding to 50% probability of fracture, T_o is the reference temperature at which fracture toughness of 1TCT specimens (B_{1T}=25.4 mm) equals to 100 MPa√m. The irradiation induced shift in T_o is also calculated.

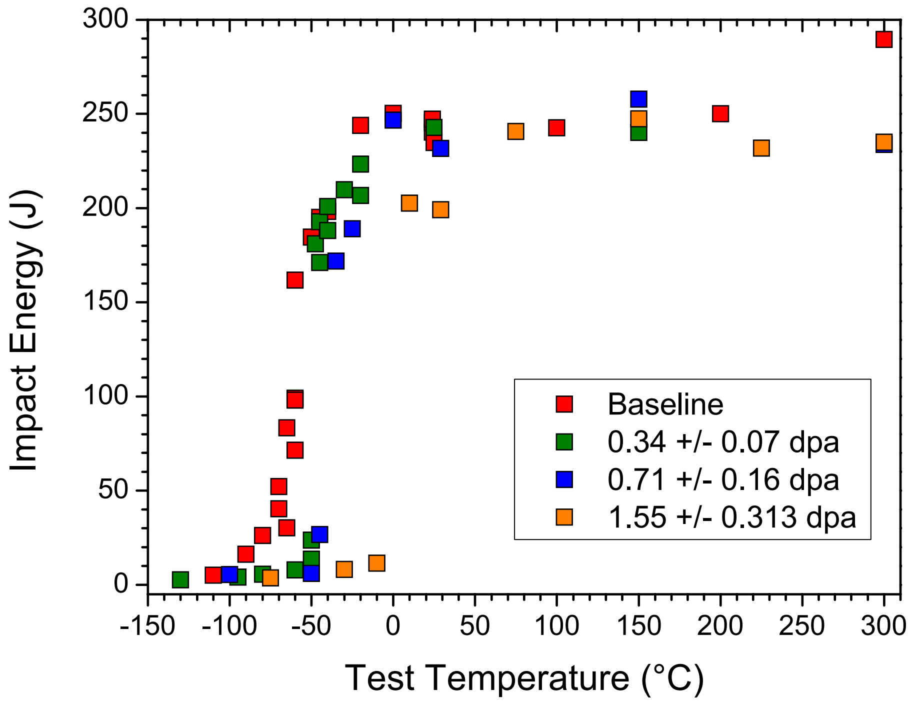

Fig. 6-2 Impact data on unirradiated and 300 °C irradiated EUROFER97 [1].

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 6-2 Results of the analyses of impact data on unirradiated and 300 °C irradiated EUROFER97 with respect to the ductile-to-brittle transition temperature (DBTT). The irradiation induced shift in DBTT is also calculated [1].

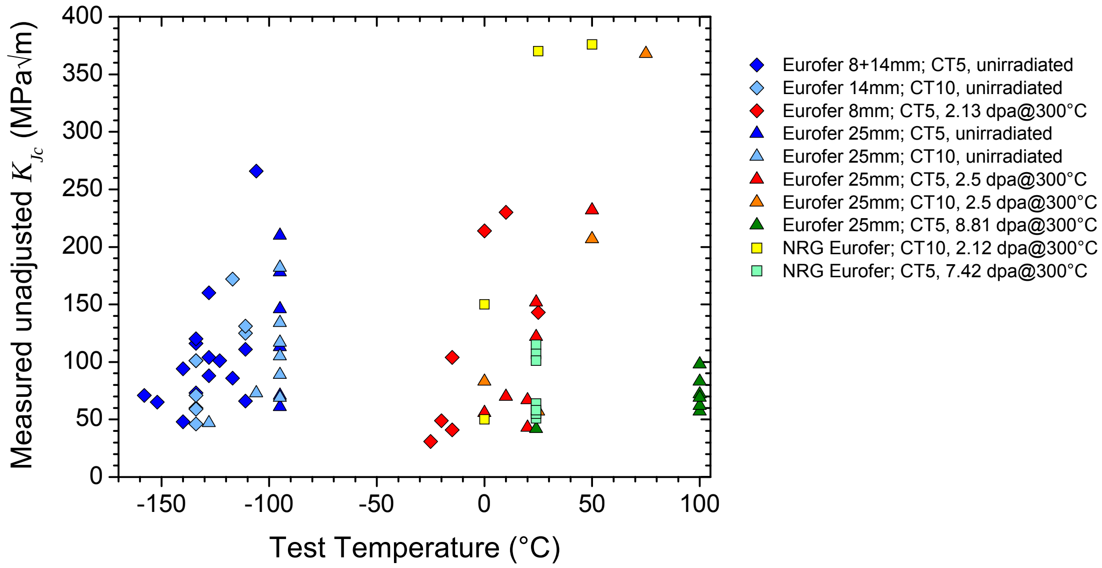

Fig. 6-3 Fracture toughness test results on unirradiated and 300 °C irradiated EUROFER97. CT5: B=5mm; CT10: B=10mm [2].

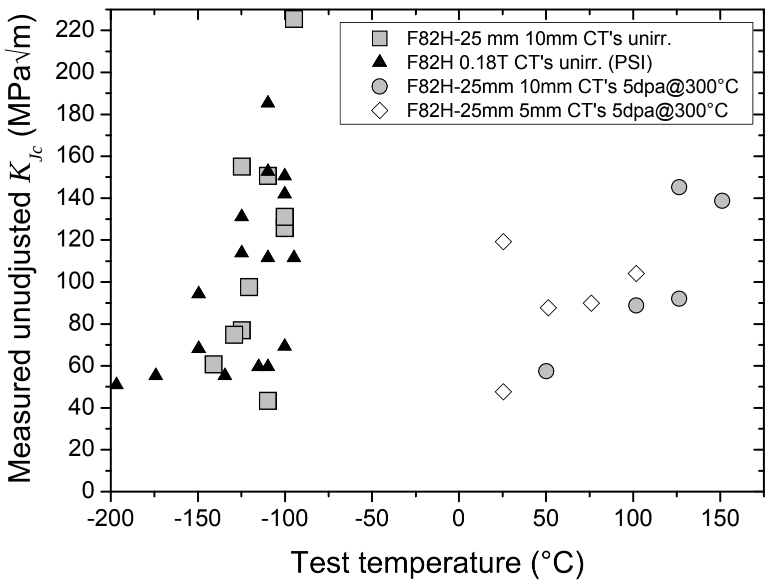

Fig. 6-4 Fracture toughness test results for unirradiated and 300 °C irradiated F82H-mod (25 mm plate) [3].

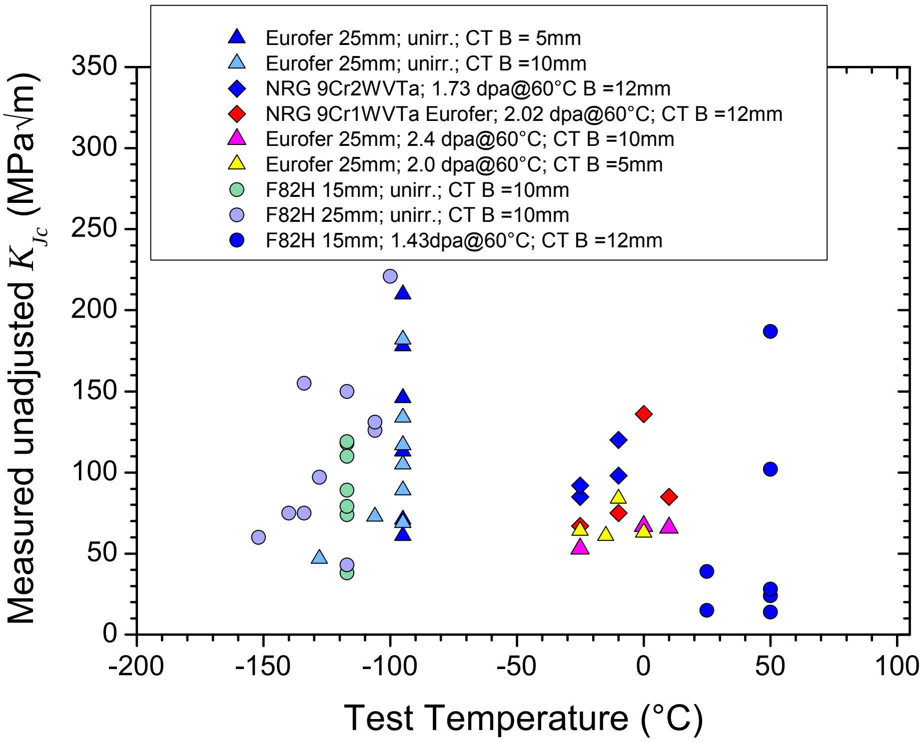

Fig. 6-5 Fracture toughness test results on unirradiated and 60 °C irradiated RAFM plate materials [2].

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 6-3 Analyses of KLST impact test on the unirradiated and irradiated EUROFER97 and other reference RAFM steels studied in [2]. The 25 mm plates of F82H have been tested in the as-received and in the optimum heat-treated (opt. HT.) conditions.

Material	Irradiation Campaign	Orientation	Specimen	Dose (dpa)	T_{irr} (°C)	T_o (°C)	Shift in T_o (°C)
F82H-IEA	unirr.	T-L	0.18 DC(T)	0	-	-65	0
F82H-IEA	HFIR RB-12J	T-L	0.18 DC(T)	3.8	349-405	-8	57
F82H-IEA	HFIR RB-11J	T-L	0.18 DC(T)	3.8	221-280	126	191
F82H-IEA	unirr.	L-T	0.18 DC(T)	0	-	-109	0
F82H-IEA	HFIR RB-12J	L-T	3.3x3.3x25.4mm	5	500	-76	33
F82H-IEA	HFIR RB-11J	L-T	3.3x3.3x25.4mm	5	300	-6	103*
F82H-HT	unirr.			0	-	-122	0

Table 6-4 Results of Master Curve analyses of fracture toughness data on the as-received (IEA 1040 °C/40min + 750C/1h) and heat treated (HT: 920 °C/1h + 750 °C/1h) F82H irradiated up to 5 dpa [4]. Fracture toughness experiments were performed on 0.18 DC(T) or on 1/3 size pre-cracked Charpy V-notched specimens ($3.3x3.3x25.4mm^3$). *No reference temperature in the unirradiated state was available for PCCV specimens in [4]. Because of this T_o determined with unirradiated DC(T) specimens was used as reference for coarse estimation of irradiation induced shift for 5dpa@300°C irradiated PCCV specimens.

Material	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-HT	3.3x3.3x25.4	0	-	14.8	-105	0
F82H-HT	3.3x3.3x25.4	5	500	12.1	-95	10
F82H-HT	3.3x3.3x25.4	5	300	11.9	5	110
F82H-HT	3.3x3.3x25.4	20	380	8.5	32	137
F82H-Std	3.3x3.3x25.4	0	-	12.3	-103	0
F82H-Std	3.3x3.3x25.4	10	300	7.9	56	159
F82H-Std	3.3x3.3x25.4	12	400	9.7	14	117
F82H-mod	3.3x3.3x25.4	0	-	10.8	-82	0
F82H-mod	3.3x3.3x25.4	9	300	8.3	70	152
F82H-mod	3.3x3.3x25.4	11	400	8.3	64	146
9Cr-2WVTa	3.3x3.3x25.4	0	-	10.8	-94	0
9Cr-2WVTa	3.3x3.3x25.4	11	400	6.5	-15	79

Table 6-5 Impact properties on F82H in the as-received (IEA 1040 °C/40min+ 750 °C/1h) and heat treated (HT: 920 °C/1h + 750 °C/1h) conditions [4]. The 1/3 size Charpy V specimens (3.3x3.3x25.4mm^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.

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	300	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

Table 6-6 Impact properties of EUROFER97 and selected RAFM steels studied in [7]-[14]. All specimens have been 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; DBTT: ductile-to-brittle-transition temperature; USE: upper shelf energy; LTUS: lowest temperature in the upper shelf.

6.2 Assessment of Fracture Mechanical Data on RAFM Steels

Material	Association	Product form	Specimen type	B (mm)	Dose (dpa)	T_{irr} (°C)	N	r	T_o (°C)
EUROFER97	NRG	8mm plate	CT	5	0	-	8	8	-112.0
EUROFER97	NRG	14mm plate	CT	5	0	-	8	7	-111.5
EUROFER97	NRG	14mm plate	CT	10	0	-	8	8	-112.3
EUROFER97	NRG	25mm plate	CT	5	0	-	6	6	-98.4
EUROFER97	NRG	25mm plate	CT	10	0	-	8	8	-92.8
EUROFER97	NRG	8mm plate	CT	5	2.13	300	7	7	-3.2
EUROFER97	NRG	25mm plate	CT	5	2.5	300	7	7	38.4
EUROFER97	NRG	25mm plate	CT	10	2.5	300	4	3	26.2
EUROFER97	NRG	25mm plate	CT	5	8.81	300	7	7	152.9
NRGEurofer	NRG	25mm plate	CT	10	2.12	300	4	2	-17.8
NRGEurofer	NRG	25mm plate	CT	5	7.42	300	7	7	62.1

Table 6-7 ASTM E1921-05 analysis of fracture toughness data on EUROFER97 [2] for determination of T_o. Product form, specimen type, specimens thickness (B), irradiation condition (dose, T_{irr}), number of performed tests (N) and number of valid tests (r) are indicated.

Material	Association	Product form	Dose (dpa)	T_{irr} (°C)	N	r	T_o (°C)	ΔT_o (°C)
EUROFER97	SCK.CEN	∅=100mm	0	-	9	7	-121.1	0
EUROFER97	SCK.CEN	∅=100mm	0.33	300	15	12	-76.6	44.5
EUROFER97	SCK.CEN	∅=100mm	0.74	300	11	9	-62.1	59
EUROFER97	SCK.CEN	∅=100mm	1.62	300	12	12	-14.5	106.6
EUROFER97	NRG	8mm plate	0	-	8	8	-112.0	0.0
EUROFER97	NRG	8mm plate	2.13	300	7	7	-3.2	108.8
EUROFER97	NRG	14mm plate	0	-	16	15	-111.9	0.0
EUROFER97	NRG	25mm plate	0	-	14	14	-95.3	0.0
EUROFER97	NRG	25mm plate	2.5	300	11	10	33.4	128.7
EUROFER97	NRG	25mm plate	8.81	300	7	7	152.9	248.2
NRGEurofer	NRG	25mm plate	2.12	300	4	2	-17.8	n.s.
NRGEurofer	NRG	25mm plate	7.42	300	7	7	62.1	n.s.
EUROFER97	NRG	25mm plate	2.17	60	7	n.s.	55.5	150.8
NRGEurofer	NRG	25mm plate	2.02	60	4	n.s.	14.6	n.s.

Table 6-8 ASTM E1921-05 analysis of fracture toughness data on EUROFER97 [1],[2] for determination of T_o. The fracture toughness data are grouped according to product form and irradiation condition (dose, T_{irr}). (n.s. stands for *not specified*). N and r are numbers of performed and valid tests, respectively.

Material	Association	Product form	Dose (dpa)	T_{irr} (°C)	N	r	T_o (°C)	ΔT_o (°C)
F82H-mod	NRG	25mm plate	0	-	10	n.s.	-114.8	0.0
F82H-mod	NRG	25mm plate	5	300	10	n.s.	83.5	198.3
F82H-mod	NRG	15mm plate	0	-	7	7	-97.5	0.0
F82H-mod	NRG	15mm plate	1.43	60	7	7	39.1	136.6

Table 6-9 ASTM E1921-05 analysis of fracture toughness data on F82H-mod [2],[3] for determination of T_o. The fracture toughness data are grouped according to product form and irradiation condition (dose, T_{irr}). (n.s. stands for *not specified*). N and r are numbers of performed and valid tests, respectively.