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Characteristics of unirradiated and 60 °C, 2.7 dpa irradiated Eurofer97

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Abstract

After the delivery of various product forms of Eurofer97, NRG has started a comprehensive characterisation effort. A laboratory-scale heat with the same specifications has been acquired as well. A 2.7 dpa irradiation has been carried out in the high flux reactor at Petten at reactor ambient temperature. Microstructural properties are investigated. Pre-irradiation as well as post-irradiation tensile, impact properties, and transition fracture toughness are determined. The microstructure of Eurofer97 is martensitic with ASTM 10.2 prior austenite grain size and few inclusions. Impact properties of the 25 mm plate are impaired: DBTT of −75 °C as opposed to −100 °C for the other forms. Tensile properties of Eurofer97 before and after irradiation are comparable to that of F82H under similar conditions. In impact tests, the Eurofer lab-scale heat outperforms F82H: the DBTT after irradiation is around −17 °C in stead of +35° for F82H-mod. Static fracture toughness tests show a larger shift in transition temperature than the impact tests.

1. Introduction

Eurofer97 [1] is the reference 9CrWVTa steel for the European long-term fusion programme in the current, fifth framework programme. It is envisaged as a structural steel for the tritium breeding blanket in a DEMO-type plant, and for blanket mock-ups in ITER, the next step fusion device. It combines moderately high service temperature (<550 °C), good post-irradiation toughness and transition temperature and post-irradiation chemical and dimensional stability. The irradiation resistance of Eurofer97 and other 9Cr-steels at low temperature is of interest for the design of a so-called International Facility for Materials Irradiation for Fusion (IFMIF), which is planned for the next decade.

During the last framework programme, a modified heat of F82H was supplied by the Japanese to the IEA [2] and has been extensively characterised in the US, Europe and Japan [3–8]. It still serves as a reference for comparing the irradiation resistance of many steels of similar composition.

2. Experimental

2.1. Materials

Eurofer97 [1] is of a nominal 9Cr–1.1W–0.2V–0.07Ta–0.1C (Fe balance) composition (all wt%). It is a fully air-hardening martensitic steel that is used in the normalised and tempered condition. 8, 14, and 25 mm thick plates and 100 mm round bar have been investigated. Normalisation has been performed at 980 °C and the temper treatment is 760 °C/1.5 h/AC for the plates and 740 °C/3.7 h/AC for the bar.

NRG ordered a laboratory-scale heat (VS3102) [9] in accordance with the Eurofer97 specification [1] in 1998 at British Steel in the UK. It is named BS-Eurofer and produced as hot-rolled 25 mm thick plate. Its

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normalising and tempering treatment has been 1050 and 750 °C/2 h/AC.

These types of steel are derived from modified high temperature 9CrMoVNb steels like P91/T91 that are used in the conventional nuclear and non-nuclear industries. The substitution of Mo for W as the main solution hardening element and Nb for Ta as the high-temperature stable carbide former makes these steels reduced activation materials in a fusion neutron environment.

2.2. Irradiation

An irradiation (code-named SIWAS-09) has been performed in a basket-type sample holder in the high flux reactor in Petten, The Netherlands, up to a calculated dose level of approximately 2.7 dpa [10]. The neutron flux is of the order of 10^{18} $\mathrm{n\,m^{-2}\,s^{-1}}$ and the irradiation lasted 150 full power days. The specimens have been in contact with the reactor pool coolant water at specimen temperatures of approximately 60 °C.

2.3. Mechanical testing

All mechanical tests have been performed in the Hot Cell Laboratory (HCL) of NRG in Petten. The tensile tests have been done in air at a strain rate of 5×10^{-4} $\mathrm{s^{-1}}$. Tensile specimens are sub-size, 4 mm diameter, 20 mm gauge length.

Impact tests are done on an instrumented miniaturised impact machine with an impact speed of 3.85 $\mathrm{m\,s^{-1}}$ with a 50J ISO striker. The impact specimens are so-called KLST specimens, $3 \times 4 \times 27$ mm of size with a 1.0 mm deep notch.

The quasi-static fracture toughness experiments have been done on plane sided, sub-size CT specimens of nominal $10 \times 27 \times 29$ mm dimensions with a W-size of 22.5 mm. The specimens are pre-cracked up to an a/W of 0.5. The toughness values are calculated in accordance with ASTM 1921 and are not corrected for loss of constraint.

3. Results

3.1. Microscopy

The microstructural characterisation has been performed at ECN in Petten. A martensitic morphology has been observed for all Eurofer97 product forms in this study with a prior austenite grain size of ASTM 10.1–10.5. For comparison, F82H-mod. was found to have a prior austenite grain size of ASTM 4. Few inclusions have been found in optical study and SEM, and they are identified as Al-oxides, FeMnS, Ta-rich and Ti-rich particles. Especially the latter is unexpected, considering the low Ti content reported by the manufacturer of 0.006–0.009 wt%. The Ta-rich particles, found in segregation-induced stringers, suggest insufficient solution treatment. The hardness is 208 ± 5 HV5, with an even distribution over the plate/bar cross-sections. No δ-ferrite is found.

BS-Eurofer shows a difference compared to Eurofer97 only in the prior austenite grain size: the higher austenitisation temperature has produced a larger prior austenite grain of ASTM 8.6 ± 0.15. All other optical and SEM investigations have produced the same observations as for Eurofer97.

3.2. Pre-irradiation mechanical tests

In Fig. 1(a) the tensile results for Eurofer97 and BS-Eurofer are compared with F82H-mod. – heats 9741 and 9753. The elastic limit and the tensile strength of Eurofer97 fall between those of the two F82H heats, but the temperature dependence of Eurofer97 is larger. The BS-Eurofer strength values fall just below the lower band of Eurofer97. The ductility of Eurofer97 and BS-Eurofer is found to be equal. No influence of orientation is observed.

In Fig. 1(b), the impact results on Eurofer97 and BS-Eurofer are shown. The ductile to brittle transition temperature (DBTT) of Eurofer97 8 and 14 mm plates and the 100 mm round bar and BS-Eurofer are not significantly different, and lies around −100 °C for the half-USE. However, for 25 mm plate a higher DBTT is found of around −75 °C. In Fig. 1(c), the tensile results of 25 mm plate are revisited and compared with the other Eurofer97 product forms. It shows firstly that there is a consistent difference of 15–40 MPa between specimens from the bottom of the plate and the top (printed side). Secondly, the yield stress is generally higher than for the other product forms while the UTS is generally lower.

3.3. Post-irradiation mechanical tests

Tensile results for irradiated Eurofer97 and BS-Eurofer are compared in Fig. 2(a) and (b). Considerable hardening has occurred during irradiation: yield stress at room temperature has increased 400 MPa and UTS has increased 190 MPa at room temperature. The lower tensile strength characteristics of BS-Eurofer remain, UTS and yield stress are 25 MPa lower than for Eurofer97, even though they have been at the highest flux positions in the irradiation rig. The irradiation hardening is comparable to that of F82H [11].

Uniform elongation decreases after irradiation to below 1%, although reduction of area remains high and total elongation therefore is still some 7%. From both Fig. 2(a) and (b) it is clear that at testing temperatures of

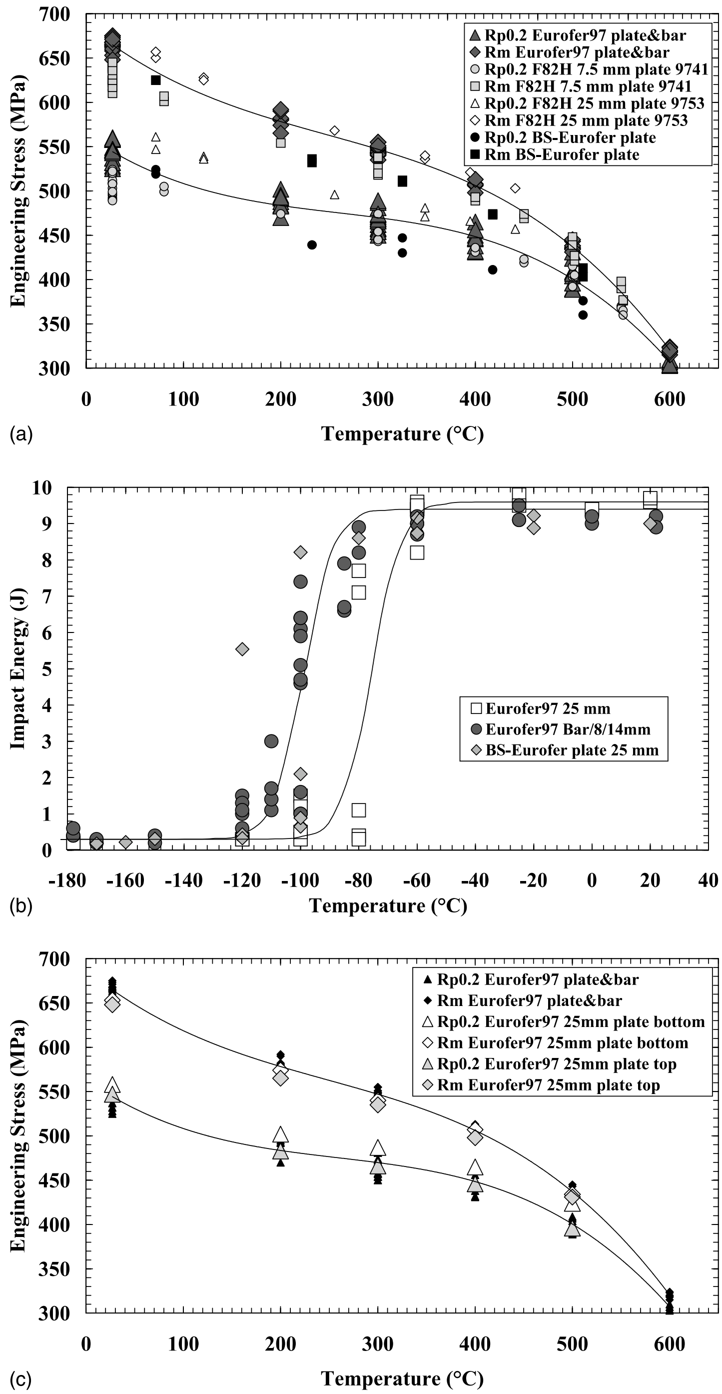
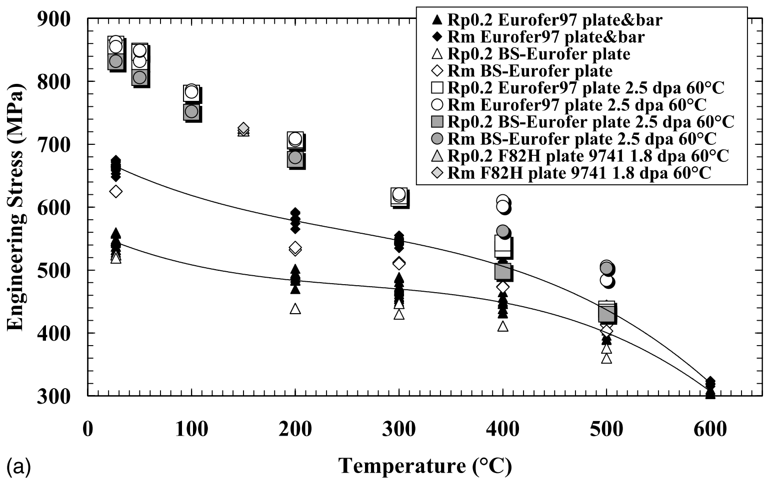


Fig. 1. (a) Tensile engineering stresses for Eurofer97, BS-Eurofer and F82H-mod. heat 9741and 9753. (b) KLST impact energies for Eurofer97 and BS-Eurofer. (c) Tensile engineering stresses for Eurofer97 25 mm plate compared to other product forms of Eurofer97.

200 °C and above there is considerable annealing of irradiation induced hardening and embrittlement.

Impact behaviour after irradiation is shown in Fig. 3. There is a shift in DBTT from approximately −100 °C to approximately −20 °C for BS-Eurofer. No Eurofer97 specimens were available for testing. F82H-mod. showed a shift to +35 °C at a 22% lower average dose level (2.1 vs. 2.7 dpa) at 60 °C. After 300 °C irradiation,



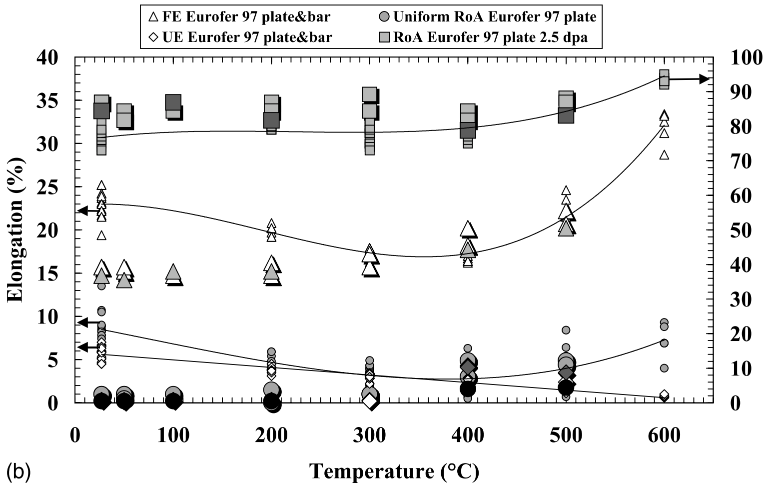


Fig. 2. (a) Tensile engineering stresses for 60 °C irradiated Eurofer97, BS-Eurofer and F82H-mod. The larger symbols refer to irradiated data. (b) Ductility for 60 °C irradiated Eurofer97. The darker symbols are BS-Eurofer, the larger symbols are irradiated data.

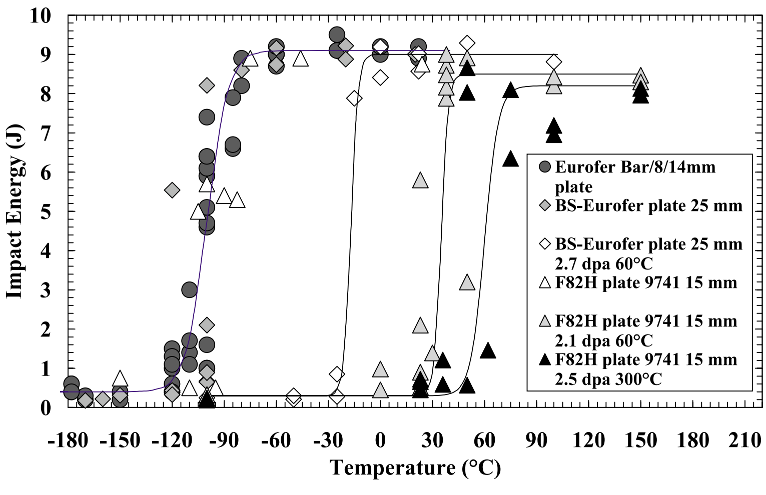


Fig. 3. KLST impact energies for 60 °C irradiated BS-Eurofer and 60 and 300 °C irradiated F82H-mod.

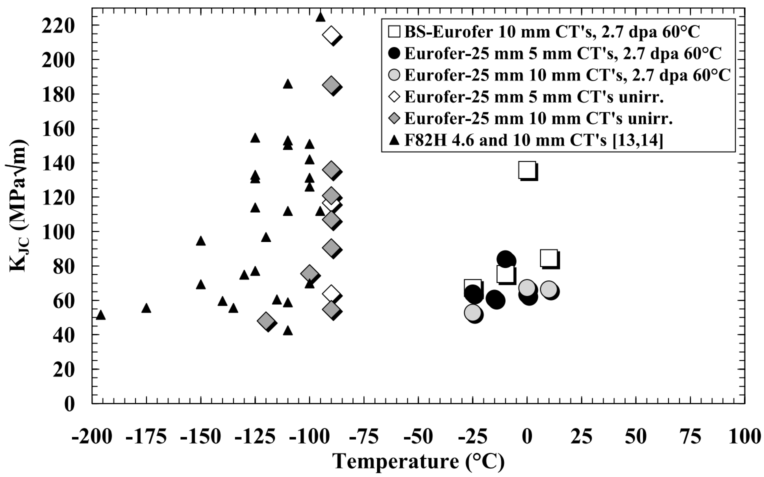


Fig. 4. Transition fracture toughness values for unirradiated F82H and unirradiated and 60 °C irradiated Eurofer97 and BS-Eurofer.

the DBTT shift of F82H-mod. is up to approximately 60 °C [12].

Transition fracture toughness results in Fig. 4 show an unirradiated T_0 at 100 MPa $\sqrt{}$m of approximately −100 °C. Irradiation has shifted the T_0 to approximately +25 °C. The unirradiated combined F82H data of Spätig [13] and Rensman [14], which fall within the same scatter band, indicate that 25 mm Eurofer plate has a higher transition temperature in the unirradiated state although the size of the data set for Eurofer is limited and prevents firm conclusions.

4. Discussion

From the optical and SEM microstructural investigations no indications are found that any of the product forms of Eurofer97 are deviant. However, the 25 mm thick plate shows a higher DBTT in miniaturised impact tests and higher yield and lower UTS in addition to sensitivity to sampling location. This leads to the conclusion that the 25 mm plate has impaired deformation capacity and toughness by microstructural origin on TEM-scale. No evidence is found for thermal-mechanical history differences or significant compositional deviation. Therefore, Eurofer97 is considered sensitive to small processing variations.

The pre-irradiation and post-irradiation tensile properties of Eurofer97 are comparable to those of F82H-mod. (Figs. 2(a) and 3). The difference in yield stress and ultimate strength with BS-Eurofer persists after irradiation, even when the latter material has received a higher dose (est. +10%) on average. The impact properties before irradiation of Eurofer97, BS-Eurofer and F82H-mod. are the same, whereas a difference of 50 °C in DBTT shift after comparable irradiations is observed between BS-Eurofer and F82H-mod., with the lowest DBTT for the former.

The shift in DBTT has been used to screen various materials for their resistance to neutron irradiation induced embrittlement. Static fracture toughness experiments indicate that they are a poor estimate of shift in the T_0 reference temperature, which is appropriate for use in designs. Dynamic impact testing with a notched specimen does not bring out the critical conditions for fast fracture in case of sharp cracks as pronounced as static fracture toughness experiments do. F82H seems to have a lower T_0 than 25 mm Eurofer plate in the unirradiated state.

5. Conclusions

There is no unambiguous relation between observations from light and SEM microscopy and the mechanical properties, even if very similar RAFM materials are studied. Although there is no indication for this from the microscopy referred to above, the 25 mm Eurofer97 plate material is deviant in tensile and impact behaviour from other product forms of Eurofer97.

A lab-scale heat BS-Eurofer was made in accordance with the specifications of Eurofer97, and very similar pre-irradiation tensile and impact properties were measured.

The pre-irradiation and post-irradiation tensile properties of Eurofer97 and BS-Eurofer are comparable to those of F82H-mod. However, the post-irradiation impact properties of the latter do not relate to those of BS-Eurofer, which are much better than those of F82H-mod.

The experiments indicate that shifts in ductile to brittle transition temperature as measured from miniaturised dynamic tests on KLST specimens underestimate the shift in the reference transition temperature T_0 in quasi-static fracture toughness measurements from CT experiments.

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References

[1] B.v.d. Schaaf et al., J. Nucl. Mater. 283–287 (2000) 52.
[2] A. Hishinuma, Y. Takagi, Data Sheets of IEA Modified F82H, Heat 9741 and Heat 9753, Handouts at IEA WG meetings in 1994/1995, JAERI & NKK, Japan.
[3] K. Shiba et al., J. Nucl. Mater. 233–237 (1996) 309.
[4] R. Schäublin et al., J. Nucl. Mater. 258–263 (1998) 1178.
[5] A.F. Rowcliffe et al., J. Nucl. Mater. 258–263 (1998) 1275.
[6] M.L. Hamilton et al., J. Nucl. Mater. 258–263 (1998) 1222.
[7] A. Alamo et al., J. Nucl. Mater. 283–287 (2000) 353.
[8] Y. Miwa et al., J. Nucl. Mater. 283–287 (2000) 334.
[9] P. Dutton, Processing of VS3102, private communication with British Steel, 1998.
[10] H. Lohner, SIWAS-09, Irradiation of Steel Specimens at Ambient Temperature – Project R268-09 Irradiation Report, JRC report P/F1/01/01, JRC Petten, 2001, The Netherlands.
[11] E.V. van Osch et al., Tensile Properties of 2.5 dpa 300 °C, Neutron Irradiated RA FM Plate, Powder HIP, and EB and TIG Welds, NRG report 26704, November 2000, Petten, The Netherlands.
[12] E.V. van Osch et al., Miniaturised Impact Properties of 2.5 dpa 300 °C, Neutron Irradiated RA FM Plate and EB and TIG Welds, NRG report 26974, October 2000, Petten, The Netherlands.
[13] P. Spätig et al., J. Nucl. Mater. 283–287 (2000) 721.
[14] J. Rensman et al., Tensile properties and transition behaviour of RAFM steel plate and welds irradiated up to 10 dpa at 300 °C, these Proceedings.