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Embrittlement behavior of neutron irradiated RAFM steels

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Abstract

The effects of neutron irradiation on the embrittlement behavior of reduced activation ferritic/martensitic (RAFM) steel EUROFER97 for different heat treatment conditions have been investigated. The irradiation to 16.3 dpa at different irradiation temperatures (250–450 °C) was carried out in the Petten High Flux Reactor in the framework of the HFR Phase-IIb (SPICE) irradiation project. Several reference RAFM steels (F82H-mod, OPTIFER-Ia, GA3X) and MANET-I were also irradiated at selected temperatures. The embrittlement behavior and hardening were investigated by instrumented Charpy-V tests with subsize specimens. The neutron irradiation induced embrittlement and hardening of as-delivered EUROFER97 are comparable to those of investigated reference steels, being mostly pronounced for 250 °C and 300 °C irradiation temperatures. Heat treatment of EUROFER97 at higher austenization temperature substantially improves the embrittlement behavior at irradiation temperatures of 250 °C and 350 °C.

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels are promising structure materials for first wall and blanket applications in fusion power plants. Mechanical properties of modified commercial (MANET) steels and newly developed RAFM (OPTIFER, F82H) steels have been thoroughly studied within the former irradiation programs (FRUST/SIENA, MANITU, HFR-Ib) up to 2.4 dpa (MANET to 15 dpa) at different irradiation temperatures (250/300/350/400/450 °C) [1–4]. Although, newly developed RAFM steels exhibit clearly better irradiation performance than the modified commercial alloys, the hardening induced by neutron irradiation accompanied by embrittlement, reduction of toughness and ductility remains a main limitation in their application, indicating the need of further material improvement.

In this work we report on the neutron irradiation induced embrittlement and hardening of the European reference steel for the first wall of a DEMO fusion reactor, EUROFER97, irradiated to 16.3 dpa at different irradiation temperatures of 250/300/350/400/450 °C. The irradiation performance of EUROFER97 is compared with the results on international reference steels F82H-mod, GA3X and OPTIFER-Ia.

2. Experimental

Subsized Charpy-V specimens of KLST type were chosen for the investigation of embrittlement

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and hardening; see [1–4] for specimen geometry. In order to enable a direct comparison with previous results, the specimens were machined parallel to the rolling direction of the steel plates (L–T-orientation).

An industrial heat of EUROFER97 steel was produced by Böhler Austria GmbH, see Table 1 for the steels' chemical composition. Part of the specimens (labeled EUROFER97 ANL) was machined from the 25 mm thick EUROFER97 plates in the as-delivered state (i.e. austenized at 980 °C, tempered at 760 °C). In order to study the influence of higher austenitizing temperature on a laboratory scale, another part of the specimens (labeled EUROFER97 WB) was machined from EUROFER97 plates subjected to a heat treatment at the higher austenitizing temperature of 1040 °C. An extensive microstructural investigation on EUROFER97 for different austenitizing temperatures was performed in [5]. For comparative purpose the heat treatments for reference alloys F82H-mod, GA3X, OPTIFER-Ia and martensitic steel MANET-I were identical to those used in previous experiments [3,4]. Table 2 lists the heat treatment conditions along with selected properties of the materials.

The specimens were irradiated in the HFR Phase-IIb–SPICE (**S**am**p**le Holder for **I**rradiation of Miniaturized Steel Spe**c**im**e**ns Simultaneously at Different Temperatures) irradiation experiment in the Petten High Flux Reactor [6]. The irradiation dose of 16.3 dpa in steel was achieved as measured by activation detectors installed in the sample holder. The irradiation temperatures of 250/300/350/400/450 °C were maintained by a balance between nuclear heating and cooling with liquid sodium. EUROFER97 ANL was irradiated at all irradiation temperatures. Because of the limited irradiation space, other materials were irradiated at selected temperatures only.

The instrumented impact tests of irradiated specimens were performed with a newly built facility installed in the hot cells of the fusion material laboratory. The facility is identical to the one used for testing of unirradiated specimens. The test and evaluation procedures are identical to those employed in previous investigations [1–4]: 25 J

Table 1
Chemical composition of materials in wt% (n.s. stands for not specified)

	EUROFER97	Reference RAFM steels			10–11% Cr–NiMoVNb
		GA3X	F82H-mod	OPTIFER-Ia	MANET-I
Heat	83697		9741	664	53645
Cr	8.91	9.17	7.7	9.33	10.8
W	1.08	2.12	2.04	0.965	n.s.
Mn	0.48	0.042	0.16	0.50	0.76
V	0.2	0.314	0.16	0.26	0.2
Ta	0.14	0.011	0.009	0.066	n.s.
C	0.12	0.159	0.09	0.10	0.14
Si	0.04	n.s.	0.11	0.06	0.37
P	0.005	n.s.	0.002	0.0046	0.005
S	0.004	n.s.	0.002	0.005	0.004
Ni	0.02	0.021	0.021	0.005	0.92
Mo	<0.001	0.0077	0.003	0.005	0.77
Nb	0.0017	0.011	0.0101	0.009	0.16
Al	0.009	0.015	0.0016	0.008	0.054
B	0.001	n.s.	0.0004	0.0062	0.0085
N	0.02	0.0018	0.008	0.0153	0.02
O	0.0008	n.s.	n.s.	n.s.	n.s.
Co	0.006	0.003	0.0037	n.s.	0.01
Cu	0.0015	0.0017	0.0063	0.035	0.015
Zr	<0.005	n.s.	0.01	n.s.	0.059
Ce	n.s.	n.s.	n.s.	<0.001	n.s.
Ti	0.006	0.001	0.004	0.007	n.s.
As	<0.005	n.s.	n.s.	0.0093	n.s.
Sb	<0.005	n.s.	n.s.	<0.0002	n.s.
Sn	<0.005	n.s.	n.s.	0.0005	n.s.
Fe	Balance	Balance	Balance	Balance	Balance

Table 2
Heat treatment and selected properties of unirradiated materials

	Heat treatment	Grain size (μm)	USE (J)	DBTT (°C)	Dynamic yield stress (MPa)
EUROFER97 ANL	980 °C/0.5 h + 760 °C/1.5 h	16 [5]	9.84	−81	543 @100 °C
EUROFER97 WB	1040 °C/0.5 h + 760 °C/1.5 h	21.4 [5]	9.84	−91	486 @100 °C
GA3X	1000 °C/1 h + 700 °C/2h	55 ± 5	9.4	−58	650 @100 °C
F82H-mod	950 °C/0.5h + 750 °C/2h	55	9.7	−86	446 @100 °C
OPTIFER-Ia	900 °C/0.5h + 780 °C/2h	10	10.6	−81	482 @23 °C
MANET-I	980 °C/2h + 1075 °C/ 0.5h + 750 °C/2h	30 ± 5	6.6	−30	670 @100 °C

pendulum impact hammer; distance between supports 22 mm; impact velocity 3.85 m/s; automatic specimen cooling, heating and transporting system; test temperature between −180 °C and 600 °C.

For each experiment, the force vs. deflection curve was recorded. The impact energy (E) was than determined by the integration of force vs. deflection curves. The impact energies were plotted vs. test temperature (T), see Fig. 1, and analyzed with respect to the characteristic values of the Charpy upper shelf energy (USE, i.e. maximum in the energy vs. temperature diagram) and the ductile-to-brittle transition temperature (DBTT). The latter was obtained by fitting the ductile-to-brittle transition region by a hyperbolic tangent function:

$$E(T) = (\mathrm{LSE} + \mathrm{USE})/2 + ((\mathrm{USE} - \mathrm{LSE})/2) \times \tanh((\mathrm{T\text{-}DBTT})/r) \quad (1)$$

with LSE being the impact energy in the lower shelf, and r the fitting parameter related to the slope of the curve in the ductile-to-brittle transition region.

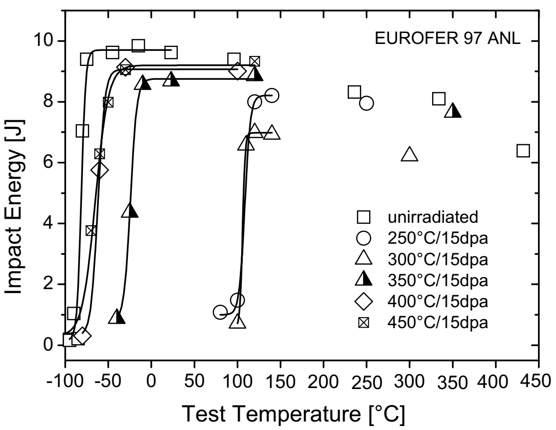

Fig. 1. Charpy impact energy vs. test temperature curves for unirradiated and irradiated EUROFER97 ANL (irradiation conditions are indicated in legend). The solid lines are fits according to Eq. (1).

Between 5 and 6 specimens for each material and each irradiation temperature ensured a sufficient number of measurement points for drawing Charpy energy vs. test temperature curves. Fig. 1 shows data for the impact properties of unirradiated and irradiated EUROFER97 ANL along with the curves fit by Eq. (1).

The dynamic yield stress (σ_{Dy}) was derived from the force vs. deflection curves at the onset of plastic deformation as stated in [1].

3. Results and discussion

Figs. 2–4 show the results obtained from the analysis of Charpy energy vs. test temperature curves.

The USE as a function of irradiation temperature (T_{irr}) is shown Fig. 2. At low irradiation temperatures ($T_{irr} \leqslant 300$ °C) the USE of EUROFER97 ANL is strongly affected by neutron irradiation, most pronounced at 300 °C. At higher irradiation temperatures ($T_{irr} \geqslant 350$ °C) the USE recovers though it still remains below the USE in the unirradiated

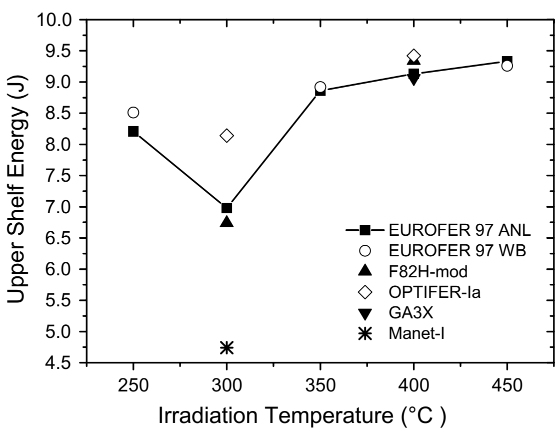

Fig. 2. Upper shelf energy vs. irradiation temperature (materials are indicated in legend).

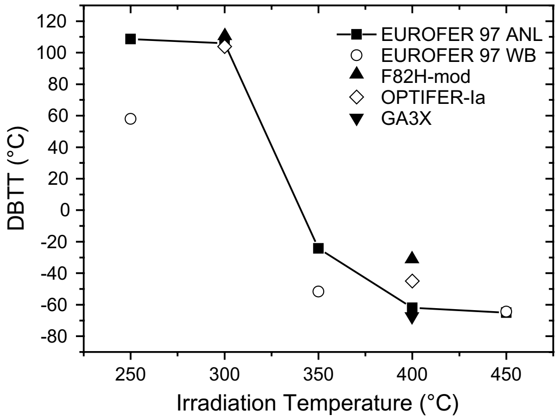

Fig. 3. Ductile-to-brittle transition temperature vs. irradiation temperature (materials are indicated in legend).

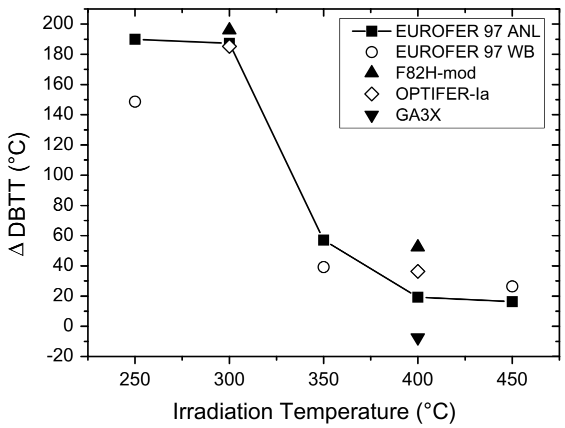

Fig. 4. Irradiation-induced shifts of ductile-to-brittle transition temperature vs. irradiation temperature (materials are indicated in legend).

condition of 9.8 J, see Table 2. The impact toughness of EUROFER97 WB are influenced by 250, 350 and 450 °C neutron irradiation in a quite similar way. The 300 °C irradiated OPTIFER-Ia shows the highest USE of 8.1 J, while the USE of 300 °C irradiated F82H-mod is comparable to that of EUROFER97 ANL. MANET-I showed the lowest impact toughness at $T_{irr} = 300$ °C. The 400 °C irradiated OPTIFER-Ia, F82H-mod and GA3X show the USE comparable to that of EUROFER97 ANL. No simple correlation can be drawn between the grain size and the USE as inferred from Table 2. The finest grain structure in the OPTIFER-Ia, however, might be an explanation for the superior USE of OPTIFER-Ia in both unirradiated and irradiated conditions.

Fig. 3 shows the DBTT vs. the irradiation temperature. For all investigated materials the DBTT is most strongly influenced at low irradiation temperatures ($T_{irr} \leqslant 300$ °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 in the DBTT between EUROFER97 materials austenized at the two different temperatures decreases with increasing T_{irr}, completely vanishing at 450 °C. The DBTT of F82H-mod and OPTIFER-Ia is comparable to that of EUROFER97 ANL at $T_{irr} = 300$ °C. However, for F82H-mod the slope of the impact energy vs. test temperature curve in transition region is smaller than the corresponding slopes for EUROFER97 ANL and OPTIFER-Ia. The DBTTs of the materials irradiated above 400 °C remain below −30 °C and thus well below the expected material application temperature. The shift in the DBTT with respect to the unirradiated state shows essentially the same behaviour with T_{irr} as seen in Fig. 4. The GA3X shows a negative shift in DBTT at $T_{irr} = 400$ °C – a behaviour already reported for 450 °C/0.8 dpa irradiation condition [3]. For boron contents below 62 wppm the influence of boron-to-helium transformation on the embrittlement cannot be resolved for 16.3 dpa irradiated steels, in clear contrast to higher boron contents and lower irradiation doses (up to 2.4 dpa) where ^{10}B burn-up was shown to contribute to the embrittlement to a large extent [3,4].

The hardening behaviour of the irradiated materials is shown in Fig. 5 where the dynamic yield stress, σ_{Dy}, is plotted vs. the irradiation temperature. The unirradiated values of σ_{Dy} are also

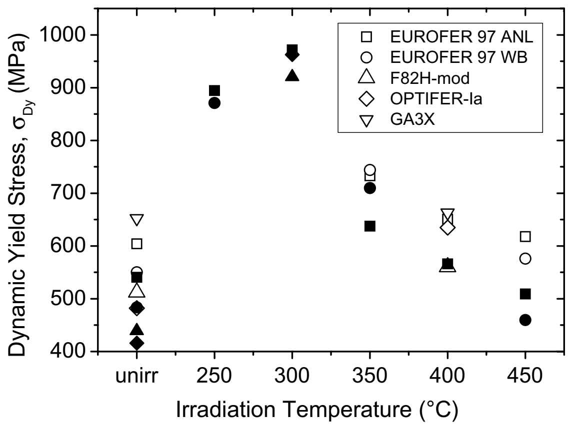

Fig. 5. Dynamic yield stress vs. irradiation temperature (materials are indicated in legend); open symbols: σ_{RT}, full symbols σ_{100}, see text for explanation.

included for comparison. Two test temperatures were used for σ_{Dy} analysis: (a) RT and (b) 100–120 °C (nominal 100 °C). At $T_{irr} \leqslant 300$ °C EUROFER97 ANL shows strong hardening ($\Delta\sigma_{Dy}$) mostly pronounced at 300 °/16.3 dpa irradiation. At $T_{irr} \geqslant 350$ °C the hardening of EUROFER97 ANL is substantially reduced in clear agreement with our previous observation on RAFM steels at lower irradiation doses up to 2.4 dpa [3,4]. EUROFER97 WB and reference RAFM steels show basically the same hardening behaviour with T_{irr}. Remarkably, at $T_{irr} = 300$ °C the dynamic yield stress is comparable for all RAFM steels, though OPTIFER-Ia shows the largest $\Delta\sigma_{Dy}$.

4. Conclusion

The European reference RAFM steel EUROFER97 irradiated to 16.3 dpa at 250–450 °C showed embrittlement behaviour and hardening comparable to that of the best reference RAFM steels. At $T_{irr} = 300$ °C the DBTT and the dynamic yield stress of EUROFER97 is comparable to that of OPTIFER-Ia. The latter showed the best impact toughness at $T_{irr} = 300$ °C. The embrittlement behaviour of F82H-mod at $T_{irr} = 300$ °C is somewhat worse than that of EUROFER97 ANL, showing large scatter in the impact energy. At $T_{irr} = 400$ °C the embrittlement of EUROFER97 is lower than that of OPTIFER-Ia and F82H-mod, though the DBTTs of these materials remain below −30 °C. A higher austenitizing temperature than the reference condition of the as-delivered EUROFER97 plate led to reduction of the embrittlement at low irradiation temperatures. The influence of boron-to-helium transformation on the embrittlement cannot be resolved for 16.3 dpa irradiated steels for boron contents below 62 wppm.

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References

[1] M. Rieth, B. Dafferner, H.-D. Röhring, J. Nucl. Mater. 233–237 (1996) 351.
[2] M. Rieth, B. Dafferner, H.-D. Röhring, J. Nucl. Mater. 258–263 (1998) 1147.
[3] H.-C. Schneider, B. Dafferner, J. Aktaa, J. Nucl. Mater. 295 (2001) 16.
[4] H.-C. Schneider, B. Dafferner, J. Aktaa, J. Nucl. Mater. 321 (2003) 135.
[5] 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 in der Helmholtz-Gemeinschaft, Wissenschaftliche Berichte FZKA 6911, October 2003.
[6] J. Ahlf, A. Zurita, High Flux Reactor (HFR) Petten – Characteristics of the Installation and the Irradiation Facilities, Nuclear Science and Technology, EUR 15151 EN, 1993.