Fusion Engineering and Design 98–99 (2015) 2038–2041

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

Tensile behavior of EUROFER ODS steel after neutron irradiation up to 16.3 dpa between 250 and 450 °C

Edeltraud Materna-Morris*, Rainer Lindau, Hans-Christian Schneider, Anton Möslang

Karlsruhe Institute of Technology KIT, Institute for Applied Materials IAM, P.O. Box 3640, D-76021 Karlsruhe, Germany

HIGHLIGHTS

- The first 9%CrWVTa steel (0.5% Y_2O_3), EUROFER ODS HIP, have been neutron irradiated up to 16.3 dpa, between 250 and 450 °C, in the High Flux Reactor (HFR).
- After post-irradiation tensile tests, there was not any increase of the upper yield strength or strain localization after irradiation which is typical of RAFM steels.
- Initially higher yield strength, $R_{p0.2}$, and distinctive tensile strength, R_m, of EUROFER ODS HIP compared to EUROFER97 steel.
- These values increased due to the neutron irradiation at lower irradiation temperatures.

ARTICLE INFO

Article history:
Received 6 October 2014
Received in revised form 3 July 2015
Accepted 14 July 2015
Available online 21 August 2015

Keywords:
RAFM steel
EUROFER97
ODS HIP
Neutron irradiation
Tensile strength

ABSTRACT

During the development of structural material for future fusion reactors, a 50 kg heat of reduced-activation ferritic-martensitic 9%CrWVTa steel with nanoscaled Y_2O_3-particles, EUROFER97 ODS HIP, was produced using powder metallurgy fabrication technology. This first batch of EUROFER97 ODS HIP and, for comparison, the steel EUROFER97 were prepared for a post-irradiation tensile test program. During neutron irradiation in the HFR (High Flux Reactor, The Netherlands), an accumulated dose of up to 16.3 dpa was reached for 771 days at full power, with the irradiation temperature ranging between 250 and 450 °C. During the post-examinations, all specimens showed the highest tensile strength at lower irradiation temperatures between 250 and 350 °C. However, ODS-alloy and steel were found to clearly differ in the mechanical behavior, which could be documented by fully instrumented tensile tests. In the un-irradiated state, tensile strength of the ODS-alloy already was increased considerably by about 60% compared to the steel. Strengthening was further increased by another 20% after neutron irradiation, but with a much better ductility than observed in the steel. The typical irradiation-induced strain localization of EUROFER97 or RAFM steels could not be observed in the EUROFER97 ODS HIP alloy.

1. Introduction

For nuclear use, it is always the aim to develop an irradiation-tolerant steel with a high strength and a high ductility. For many years now, the RAFM (reduced-activation ferritic-martensitic) 8–10%Cr-steels have been the main candidate for structural applications in future fusion reactors, such as the first wall and breeder blanket of the DEMO fusion reactor. For this purpose, a good thermo physical and mechanical behavior, low neutron irradiation-induced swelling, and compatibility with major cooling and breeding materials are required [1].

Within the European Union, the two major breeding blanket concepts developed are the helium-cooled pebble bed (HCPB) and the helium-cooled lithium lead (HCLL) blankets. For both concepts, different conceptual designs are being discussed with temperature windows in the range of 250–550 °C for conservative approaches based on RAFM steels. If higher peak operational temperatures up to 650 °C, are considered as in the plasma near surface of the HCLL concept, the use of oxide dispersion-strengthened (ODS) alloys is inevitable. The hipped ODS alloy presented in this study can be considered as the pre-cursor material for the plate material needed [2–5].

2. Experimental

The chemical compositions of the European RAFM steel EUROFER97 and its ODS version are given in Table 1. The ODS variation

* Corresponding author.
E-mail address: edeltraud.materna-morris@kit.edu (E. Materna-Morris).

http://dx.doi.org/10.1016/j.fusengdes.2015.07.015

Table 1
Alloying elements of the Fe-based test alloys in wt.%.

Heat	C	Si	Mn	Cr	V	W	N	Ta	B
EUROFER97 ODS HIP +0.5% Y_2O_3	0.072	0.115	0.418	9.4	0.185	1.10	0.029	0.08	0.005
EUROFER97 as rec.	0.12	0.04	0.48	8.91	0.2	1.08	0.02	0.14	<0.001

of the specified EUROFER97 was added 0.5% Y_2O_3. The production process included inert gas atomization of EUROFER97 (H. C. STARCK) and subsequent mechanical alloying in industrial ball mills of attritor type by PLANSEE, Austria. Following the idea to fabricate near-net-shape blanket structures by hipping, these first attempts included hot isostatic pressing (HIP) as consolidation process for the production of 4 bars of 60 mm in diameter and 300 mm in length [3]. HIP was the last treatment of the ODS material. The comparison material, EUROFER97 as rec., was used in the as received condition: 980 °C 0.5 h + 760 °C 1.5 h (Table 2).

Cylindrical tensile specimens with a total length of 38 mm, a gauge length of 18 mm, and a diameter of 3 mm were fabricated of EUROFER97 and the ODS HIP alloy. The specimens of EUROFER97 steel were taken from a plate parallel to the rolling direction and subjected to a heat treatment. The as received material of EUROFER97 ODS HIP were round bars which were furnace-cooled after HIP at a temperature of 1150 °C/2 h, 100 MPa. The specimens were also taken in longitudinal direction.

A special wrapper with irradiation capsules accommodating the tensile together with impact and fatigue specimens was inserted into the central part of the HFR reactor core. An accumulated neutron dose of up to 16.3 dpa was reached for 771 days at full power. The irradiation was always set to 250, 300, 350, 400, and 450 °C. Each position was occupied with two specimens. Temperatures were controlled by changing the gas mixture (helium and neon) in the gas gaps surrounding the samples. The cumulative neutron fluence at E > 0.1 MeV was $22.85 \times 10^{25}\ m^{-2}$. The experimentally obtained average damage level for monitor-set positions in the specimen holder was 16.3 dpa for stainless steel.

After all irradiations, the specimens were transported to the hot cells of the FML (Fusion Material Laboratory) at KIT (Karlsruhe Institute of Technology) to perform the post-irradiation tests. The tensile specimens were tested by using of a universal testing machine and the data measured were plotted stress/strain diagrams in a fully instrumented procedure. The test temperatures corresponded to the irradiation temperatures, some specimens, however, were tested at RT (room temperature, 20 °C). Selected specimens were further investigated by hardness measurements and SEM (scanning electron microscopy) to determine the irradiation-induced changes of the materials.

3. Results and discussion

Stress vs. strain curves were recorded for each experiment and documentation revealed the narrow scatter of the repeated test series. The diagrams of EUROFER97 ODS HIP exhibited a classical stress/strain behavior comparable to that of construction steels (Fig. 1). In the un-irradiated state, yield strength, $R_{p0.2}$, was 770 MPa at 20 °C. This value of $R_{p0.2}$ was 220 MPa higher than observed for EUROFER97 steel (Fig. 2). Tensile strength, R_m, of ODS increased up to 1012 MPa and that of EUROFER97 steel reached up to 651 MPa.

After neutron irradiation, all test specimens showed the highest increase in yield strength, $R_{p0.2}$, and tensile strength, R_m, in the lower temperature range between 250 and 350 °C, as was reported in [6,7] and also observed in other laboratories, e.g. [8–12]. During the tensile tests of EUROFER ODS HIP, a significant irradiation-induced yield strength increase, $\Delta\sigma_{irr}$ of about 260 MPa at 250–300 °C irradiation temperature, was observed. Comparison with EUROFER97 steel showed nearly the same level in $R_{p0.2}$, but with a much higher R_m. R_m of EUROFER ODS HIP was always higher, i.e. about 1000–1100 MPa at 250–300 °C irradiation and test-temperature. Neutron irradiation of EUROFER97 steel caused an increase in yield strength with a subsequent stress drop or so-called strain localization. This is a typical neutron irradiation-induced behavior for bcc RAFM steels due to their microstructural

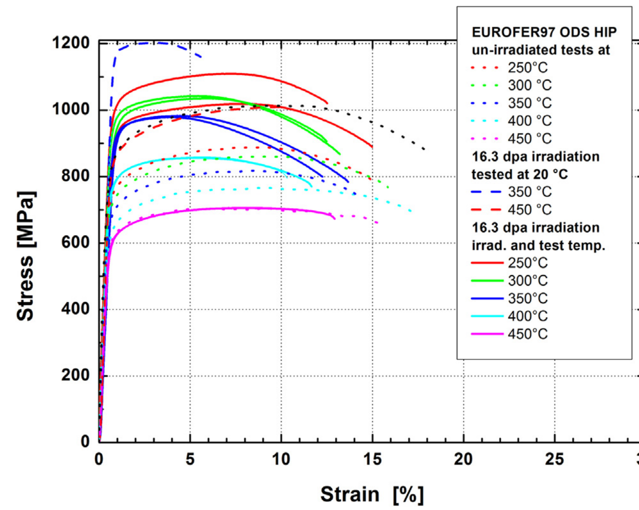

Fig. 1. Stress/strain diagrams of EUROFER97 ODS HIP.

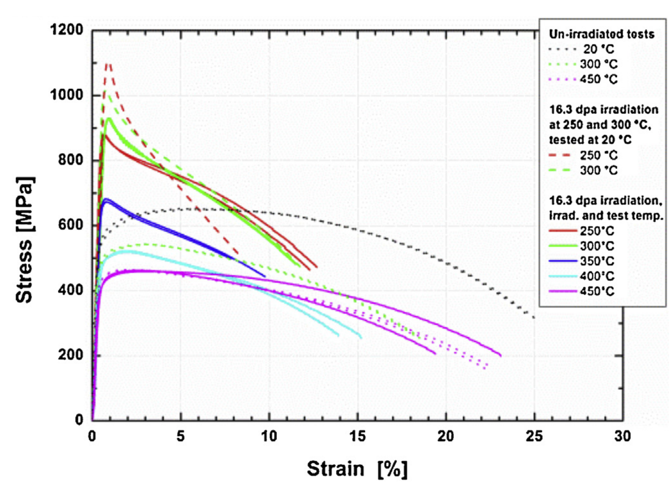

Fig. 2. Stress/strain diagrams of EUROFER97 steel.

Table 2
Material conditions.

Heat	Heat treatment
EUROFER97 ODS HIP +0.5% Y_2O_3	Not heat treated after HIP
EUROFER97 as rec.	980 °C 0.5 h + 760 °C 1.5 h

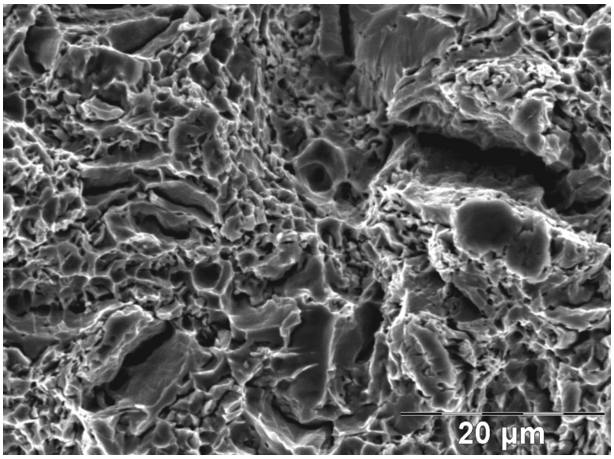

Fig. 3. Fracture surface of EUROFER97 ODS HIP, 16.3 dpa, $T_{irrad} = T_{test} = 300\,^{\circ}C$, SEM image.

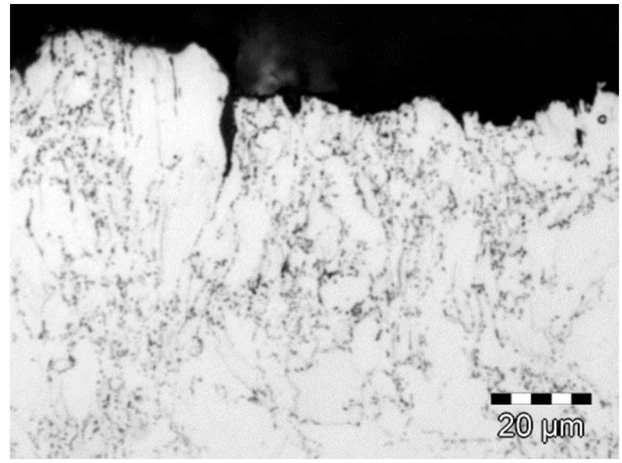

Fig. 4. Middle length cut of fracture Fig. 3, LM image.

changes, such as e.g. dislocation loops or small precipitates as interstitial atoms which are stable at lower irradiation temperatures [13]. This behavior was already observed by G. P. Seidel, 1968, in bcc Fe single crystals after neutron irradiation.

Former post-irradiation experiments of ODS materials were reported by Lucon [10]. At lower doses of about 1.6 dpa, 300 °C, strain localization was found in ODS material, too. Perhaps, the reduction and disappearance of strain localization are dependent on higher dose-levels and/or irradiation time.

At 450 °C, both materials showed nearly the same behavior, due to a minor thermal annealing effect, but again with a higher strength of the ODS material. Some tests of irradiated materials were performed at 20 °C, too. Both irradiated materials exhibited a further increase in strength.

Hardness tests HV 0.1 were performed to confirm irradiation induced strengthening by hardening (Table 3). A Δ HV 0.1 of about 80 was observed in EUROFER HIP ODS and 100 in EUROFER97 steel. At temperatures at 450 °C, no difference to the un-irradiated material was found.

Fractographic investigations were done by SEM (scanning electron microscopy). The fracture surfaces of EUROFER97 ODS HIP were covered mostly by ductile dimple formations with cracks in between (Fig. 3). Such secondary cracks were found when analyzing a middle length cut by LM (light microscopy), too (Fig. 4). These cracks developed along grain boundaries which were oriented parallel to the gauge length or tensile load. Isolated large pores could be found, too.

The EUROFER97 steel specimens were always broken in a ductile manner and the fracture surfaces showed a homogeneous transgranular dimple formation (Figs. 5 and 6).

The different material behavior during static tensile tests of EUROFER97 ODS HIP and EUROFER97 steel can be explained by their different structures. The classical explanation of the material behavior of this ODS alloy are the very small grains, which caused a higher hardness and strength than in steel. Further irradiation induced hardening by dislocation loops and point defects occurred

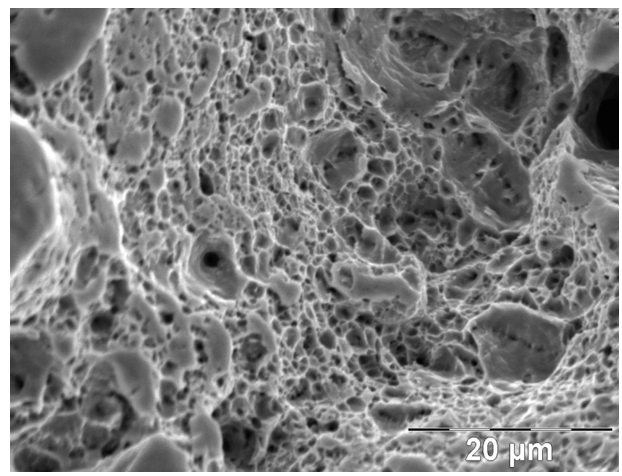

Fig. 5. Fracture surface of EUROFER97, 16.3 dpa, $T_{irrad} = T_{test} = 300\,^{\circ}C$, SEM image.

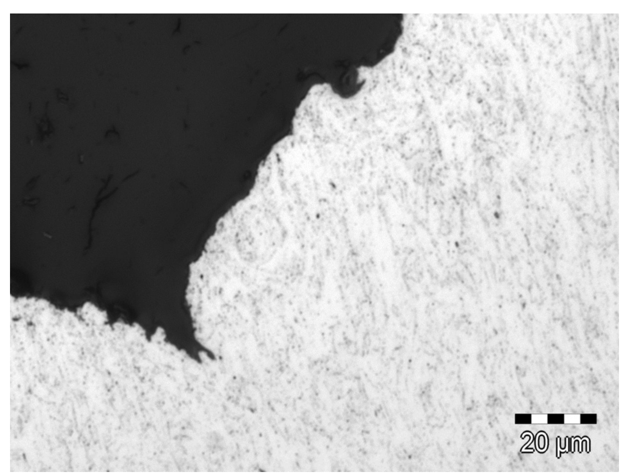

Fig. 6. Middle length cut of fracture Fig. 5, LM image.

Table 3
Hardness HV 0.1 before and after neutron-irradiation 16.3 dpa.

Heat	Un-irradiated	Irradiation temperature in °C				
		250	300	350	400	450
EUROFER97 ODS HIP +0.5% Y_2O_3	349	430	–	421	–	347
EUROFER97 as rec.	220	316	343	273	238	230

further increase in hardness and strength. First TEM-observations showed that loops and Y_2O_3-partikles have nearly the same dimension of about ∅ 10 nm at lower irradiation temperatures <350 °C (M. Klimenkov). Probably, loops and Y_2O_3-partikles are in interaction for increasing strength and a relatively good ductility. This effect could not be found in the EUROFER97 steel. This irradiation induced dislocation loops and point defect block the mobility of the dislocations with the result of decreasing ductility [14]. For future, further microstrutural investigations by TEM of EUROFER97 ODS HIP and EUROFER97 steel are necessary to understand better the mechanism of the different microstructures.

4. Conclusion

Initially higher hardness in EUROFER97 ODS HIP than in EUROFER97 steel. Hardness increased during irradiation. At the highest irradiation temperature of 450 °C, hardness of the un-irradiated level was determined. This is a typical thermal effect, all irradiation-induced hardening effects disappeared again.

Initially higher yield strength, $R_{p0.2}$, and distinctive tensile strength, R_m, of EUROFER ODS HIP compared to EUROFER97 steel. These values increased due to the neutron irradiation at lower irradiation temperatures.

The tensile stress vs. strain diagrams of EUROFER97 ODS HIP did not show any increase of the upper yield strength or strain localization after irradiation which typical RAFM steels.

These good results are remarkable for the first EUROFER97 ODS HIP. Based on the data measured, this type of material will be further developed.

Acknowledgments

This work, supported by the European Communities under the Contract of Association between EURATOM and KIT (Karlsruhe Institute of Technology), was carried out within the framework of the European Fusion Development Agreement.

References

[1] K. Ehrlich, Materials research towards a fusion reactor, Fusion Eng. Des. 56–57 (2001) 71–82.
[2] R. Lindau, A. Möslang, M. Schirra, P. Schlossmacher, M. Klimenkov, Mechanical and microstructural properties of a hipped RAFM ODS-steel, J. Nucl. Mater. 307–311 (2002) 769–772.
[3] E. Lucon, Mechanical tests on two batches of oxide dispersion strengthened RAFM steel (EUROFER97), Fusion Eng. Des. 61–62 (2002) 683–689.
[4] R. Lindau, M. Klimiankou, A. Möslang, M. Rieth, B. Schedler, J. Schröder, A. Schweiger, ODS-EUROFER – a reduced activation ferritic martensitic ODS-steel for structural applications in future nuclear fusion reactors, in: G. Kneringer, et al. (Eds.), Proceedings of 16th International Plansee Seminar, Reutte, 2005, pp. 545–557.
[5] R. Lindau, et al., Present development status of EUROFER and ODS-EUROFER for application in blanket concepts, Fusion Eng. Des. 75–79 (2005) 989–996.
[6] E. Materna-Morris, H.-C. Schneider, A. Möslang, Tensile and low cycle fatigue properties of EUROFER97-steel after 16.3 dpa neutron irradiation at 523, 623 and 723 K, J. Nucl. Mater. 442 (2013) 562–566.
[7] E. Materna-Morris, H.-C. Schneider, A. Möslang, Tensile Behavior of RAFM Alloys after Neutron Irradiation of up to 16.3 dpa between 250 and 450 °C, J. Nucl. Mater. (2014).
[8] T. Yamamoto, G.R. Odette, H. Kishimoto, J.-W. Rensma, P. Miao, On the effect of irradiation and helium on the yield stress changes and hardening and non-hardening embrittlement of ∼8Cr tempered martensitic steels: compilation and analysis of existing data, J. Nucl. Mater. 356 (2006) 27–49.
[9] E. Lucon, W. Vandermeulen, Overview of the tensile properties of EUROFER in the non-irradiated and irradiated conditions, J. Nucl. Mater. 386–388 (2009) 254–256.
[10] E. Lucon, Mechanical properties of two improved EUROFER ODS steels before and after low dose neutron irradiation at 300 °C, Fusion Sci. Technol. 56 (2009) 289–294.
[11] N. Baluc, et al., Review of the EFDA work programme on nano-structured ODS RAF steels, J. Nucl. Mater. 417 (2011) 149–153.
[12] H. Tanigawa, R.L. Klueh, N. Hashimoto, M.A. Sokolov, Hardening mechanisms of reduced activation ferritic/martensitic steels irradiated at 300 °C, J. Nucl. Mater. 386–388 (2009) 231–235.
[13] G.P. Seidel, Einfluß von Neutronenbestrahlung auf die plastische Verformung von Eisen-Einkristallen mit unterschiedlichem Kohlenstoffgehalt, Phys. Stat. Sol. 25 (1968) 175.
[14] E. Materna-Morris, A. Möslang, R. Rolli, H.-C. Schneider, Effect of helium on tensile properties and microstructure in 9%Cr-WVTa-steel after neutron irradiation up to 15 dpa between 250 and 450 °C, J. Nucl. Mater. 386–388 (2009) 422–425.