Low Cycle Fatigue Properties of Reduced Activation Ferritic/Martensitic Steels after High Dose Neutron Irradiation

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Abstract. The development and thorough characterization of DEMO relevant structural materials as well as their validation under fusion relevant conditions are the prerequisites for reliable design and safe and successful operation of the DEMO and belong to the key tasks within the European long term fusion R&D programme. The current work focuses on the Low Cycle Fatigue (LCF) behaviour of RAFM steels irradiated to a displacement damage doses up to 70 dpa at 330-337 °C in the experimental fast reactor Bor 60 within the ARBOR 2 irradiation programme. The influence of the neutron irradiation on the fatigue behaviour was determined for the as-received EUROFER97 (980 °C/0.5 h + 760 °C/1.5 h), pre-irradiation heat treated EUROFER97 HT (1040 °C/0.5 h + 760 °C/1.5 h) and pre-irradiation heat treated F82H-mod (1040 °C/38 min + 750 °C/2 h) steels. The strain controlled push-pull loading was performed with miniaturized cylindrical specimens at a constant temperature of 330 °C with different total strain ranges ($\Delta\varepsilon_{tot}$) between 0.8 and 1.1 % and at common strain rate of $3x10^{-3}$ s^{-1}. The comparison with the corresponding results in the reference unirradiated state was performed for both the adequate total and inelastic strain amplitudes. The neutron irradiation induced hardening can affect differently the fatigue behaviour of the irradiated specimens. The reduction of the inelastic strain in the irradiated state compared to the reference unirradiated state for common total strain amplitudes can yield increase of fatigue lifetime. The increase of the stress for the adequate inelastic strain in contrast might accelerate the fatigue damage accumulation. Depending upon which of the two mentioned effects is dominant the neutron irradiation can either extend or reduce the fatigue lifetime compared to the reference unirradiated state. The experimental results on EUROFER97 and EUROFER97 HT support the above considerations. Most of the irradiated specimens show fatigue lifetimes which are comparable to the reference unirradiated state for adequate inelastic strains. In some cases, however, lifetime reduction is observable. F82H mod showed partly lifetime enhancement compared to the reference unirradiated state for adequate inelastic strains.

1. Introduction

Structural materials for in-vessel components of a future fusion energy generation Demonstration Power Plant (DEMO) will be exposed to high neutron and thermo-mechanical loads. Synergistic effects of displacement damage and helium to be generated in a fusion reactor are believed to strongly influence structural materials performance. The development and thorough characterization of DEMO relevant structural materials as well as their validation under fusion relevant conditions are, thus, the prerequisites for reliable design and safe and successful operation of the DEMO and belong to the key tasks within the European long term fusion R&D programme.

The Reduced Activation Ferritic/Martensitic (RAFM) steels are considered as primary candidate structural materials for the First Wall (FW) and helium cooled Breeding Blanket (BB) with operating temperatures between 250 and 550 °C [1]. Reduced activation 9%Cr-WVTa steel EUROFER97 emerged in the course of the European application driven material development programme represents the European reference structural material for FW and BB of DEMO. A large characterization program is being performed including microstructural, mechanical and corrosion experiments. Since an irradiation facility with a fusion relevant neutron spectrum is not yet available, the irradiation performance of EUROFER97 and other international RAFM steels was extensively studied in various Material Test Reactors, see *e.g.* [2] and references therein.

The current work focuses on the Low Cycle Fatigue (LCF) behaviour of the European reference steel for the FW of a DEMO fusion reactor, EUROFER97 and selected RAFM steels after irradiation to displacement damage doses up to 70 dpa at 330-337 °C in the experimental fast reactor Bor 60 within the ARBOR 2 irradiation programme.

2. Materials

An industrial batch of the European RAFM steel EUROFER97 (nominal composition Fe-9Cr-1.1W-0.2V-0.12Ta, see *e.g.* [3]) was produced by Böhler Austria GmbH. Four different product forms: plates, with thicknesses of 8, 14, 25 mm and bars with diameter of 100 mm were distributed by Karlsruher Institut für Technologie (KIT) (formerly Forschungszentrum Karlsruhe - FZK) to different European associations. For the two ARBOR irradiations part of the specimens (referred to as EUROFER97) was machined from 25 mm thick EUROFER97 plates in the as-delivered state (980 °C/0.5 h + 760 °C/1.5 h). Another part of the specimens (referred to as EUROFER97 HT) was machined from 25 mm thick EUROFER97 plates subjected to a pre-irradiation Heat Treatment (HT) (1040 °C/38 min + 750 °C/2 h).

A 5-ton heat of modified F82H (F82H-mod, Fe-7.5Cr-2W-0.15V-0.02Ta-0.1C) was produced by NKK Corporation for collaborative research coordinated by an International Energy Agency (IEA) committee. 7.5, 15 and 25 mm plates were distributed by IEA and subsequently by KIT to the European partners. For the ARBOR irradiations the specimens were machined from the 25 mm plate subjected to a HT of 1040 °C/38 min + 750 °C/2 h.

3. Irradiation Experiment

The BOR 60 experimental fast reactor of Joint Stock Company “State Scientific Center - Research Institute of Atomic Reactors” (JSC “SSC RIAR”) offers different irradiation positions in the reactor core of 450 mm height and 550 mm diameter [4]. Within the ARBOR 1 irradiation [5] 150 mini-tensile/ LCF specimens and 150 mini-impact (KLST) specimens of nine different RAFM steels have been irradiated in a fast neutron (>0.1 MeV) flux of 1.8×10^{19} n $m^{-2}s^{-1}$ at temperatures between 331 and 338 °C up to ~30 dpa. For the study of the high dose irradiation performance of EUROFER97 and other RAFM steels, 50 % of the specimens from the ARBOR 1 irradiation were reloaded into the ARBOR 2 rig for further irradiation to a target dose of 70 dpa at 330-340 °C. The irradiation rig instrumented with temperature and neutron monitors was loaded into the instrumented cell D-23 of BOR 60 allowing direct temperature measurement during the first campaign. The calculation of the damage dose for ferritic steel specimens was conducted using the SPECTER code [6]. A cumulative damage dose up to 70 dpa was achieved at 330-340 °C.

Within the ARBOR 2 irradiation programme KIT irradiated 144 mini-tensile/ LCF and 124 Charpy impact specimens of eleven different RAFM steels.

4. Post Irradiation Examination

The post irradiation mechanical testing of the specimens from the ARBOR 2 irradiation is performed at the material science laboratory of SSC RIAR under the International Science and Technology Center (ISTC) Partner Contract Nr. # 2781p.

Cylindrical specimens of 7.6 mm gauge length and 2 mm diameter complying Small Specimen Testing Technology (SSTT) were machined in the Longitudinal-Transverse (L-T) orientation for the investigation of tensile and LCF properties [7]. LCF tests are performed with an electro-mechanical testing machine, equipped with a three-zone furnace up to 1000 °C and a high-temperature extensometer. The strain controlled push-pull (LCF) loading was performed at a constant temperature of 330 °C for different total strain ranges ($\Delta\varepsilon_{tot}$) between 0.8 and 1.2 % and at common strain rate of $3x10^{-3}$ s^{-1}. The number of cycles to failure (N_f) was defined at a point where the peak tensile stress within a cycle decreased by 30% from its value at a point marking the termination of the linear dependence of peak tensile stress on the number of cycles (N). In addition, inelastic strain ranges ($\Delta\varepsilon_{inelastic}$) at $N_f/2$ were determined for given total strain ranges from the recorded hysteresis loops. Fractography of selected specimens were investigated by Scanning Electron Microscopy (SEM).

5. Results

FIG. 1 shows total ($\Delta\varepsilon_{tot}$) and inelastic ($\Delta\varepsilon_{inelastic}$) strain ranges vs. the number of cycles to failure (N_f) in double logarithmic scale for EUROFER97 in the unirradiated condition and after neutron irradiation in ARBOR 2 programme up to 71 dpa at 330-337 °C. For comparison 31 dpa data from ARBOR 1 [7] as well as 2 dpa data from SOSIA-02 (Nuclear Research and consultancy Group - NRG) [8] are also included. In SOSIA-02 programme LCF specimens of 3 mm diameter and 7.5 mm gauge length were irradiated at 300 °C. Afterwards, those specimens were tested at 300 °C. Comparison of the results obtained in the reference unirradiated state by using SSTT (KIT) and larger (NRG) specimens indicates considerable underestimation of the fatigue lifetime by SSTT. The dashed dotted line in *FIG. 1* is reproduced from [9, 10] and represents model prediction of lifetime in reference unirradiated state at a test temperature of 330 °C. The model gives a right bound of the lifetime results obtained in the unirradiated state in [8]. In case of SSTT type specimens the neutron irradiation of EUROFER97 has only a minor influence on the fatigue behaviour for the total strain amplitudes between 0.8 and 1.1 %. In some cases a slight enhancement of the fatigue lifetime in comparison to the unirradiated state is observed. Unfortunately, the limited number of available irradiated specimens does not allow detailed statistical analysis. The comparison of irradiated and unirradiated cyclically loaded SSTT specimens in terms of the adequate inelastic strain amplitudes leads to ambiguous results. The number of cycles to failure of 47 and 70 dpa irradiated specimens remained nearly unchanged compared to unirradiated

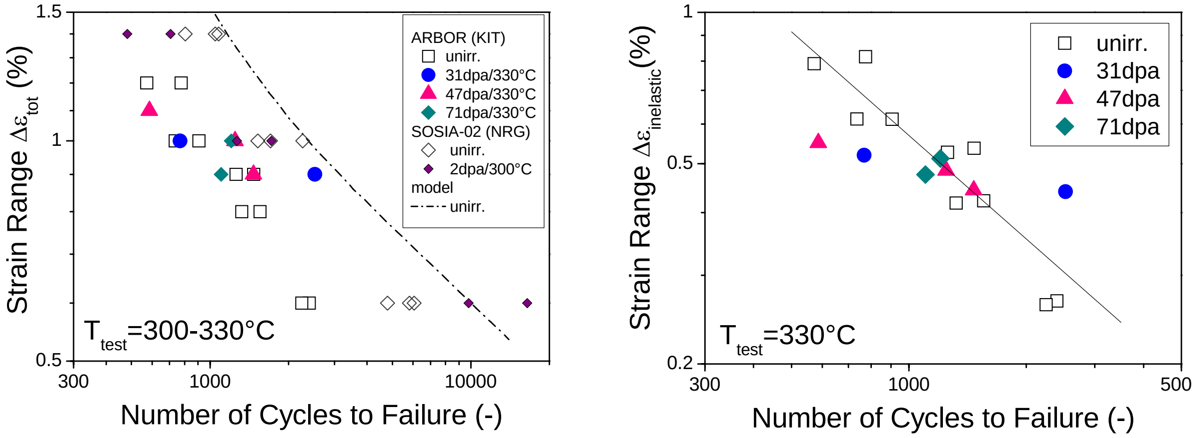


FIG. 1. Fatigue lifetime for unirradiated and up to 71 dpa irradiated (T_{irr} = 300-337 °C) EUROFER97 vs. total strain range (left) and inelastic strain range (right); The dashed line represents the model description of the unirradiated data [9, 10]. The solid line represents the description of the unirradiated data by a Manson-Coffin relation.

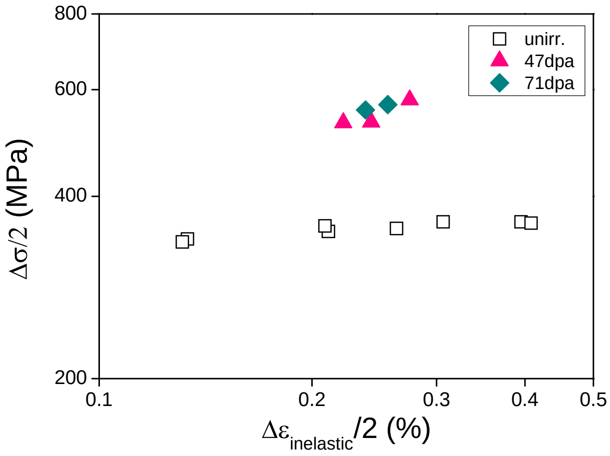


FIG. 2. Half stress span vs. half inelastic strain range determined from hysteresis loop at $N_f/2$ for unirradiated and irradiated EUROFER97.

condition for inelastic strain ranges below 0.5 %, whereas for inelastic strain ranges above 0.5 % a noticeable reduction of the fatigue life was recognized. The detailed analysis of the hysteresis loops of a specimen irradiated to 47 dpa and tested at $\Delta\varepsilon_{tot}$ =1.1 % ($\Delta\varepsilon_{inelastic}$ = 0.55 % at $N_f/2$) indicated inelastic deformation already after the first quarter cycle. The reduction of the fatigue life in irradiated specimens at high strain amplitudes might be thus related to the accumulation of fatigue damage in few activated narrow slip bands which become cleaned from radiation defects e.g. dislocation loops by moving dislocations. In addition irradiation enhanced stresses compared to the unirradiated state for adequate inelastic strain, as shown in *FIG. 2*, might also accelerate the fatigue damage accumulation. The enhancement of the fatigue lifetime in comparison with unirradiated state for low inelastic strain ranges ($\Delta\varepsilon_{inelastic}\leq 0.4$ %) is due to irradiation induced hardening.

The fatigue behaviour of EUROFER97 HT in the unirradiated condition and after neutron irradiation up to 71 dpa at 330-337 °C is shown in *FIG. 3*. The neutron irradiation has qualitatively different influence trends for 47 and 71 dpa irradiated EUROFER97 HT for total strain amplitudes between 0.8 and 1.1%. The 47 dpa irradiated specimens show if any only slight decrease of the lifetime in comparison to the unirradiated state. The 71 dpa irradiated specimens show in contrast an increase of the lifetime for adequate total strain amplitudes which is more pronounced for low strains. In terms of the adequate inelastic strains 47 dpa specimens show a decrease of the lifetime compared to the unirradiated state, whereas the lifetime of the 71 dpa specimens remains nearly unchanged. The apparent increase of the lifetime for 71 dpa specimens is thus mainly related to the irradiation induced hardening.

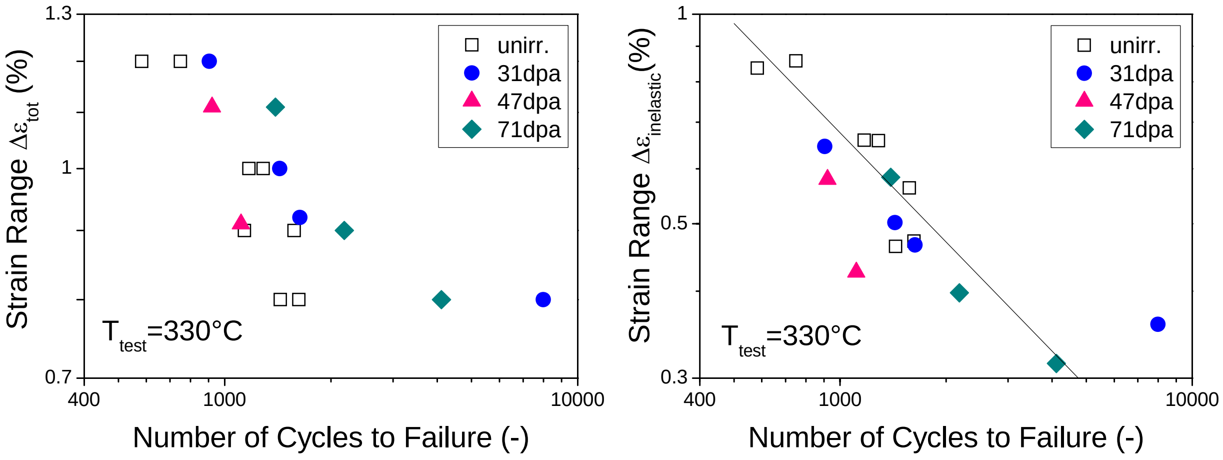


FIG. 3. Fatigue lifetime for unirradiated and up to 71 dpa irradiated (T_{irr} = 330-337 °C) EUROFER97 HT vs. total strain range (left) and inelastic strain range (right); The line represents the description of the unirradiated data by a Manson-Coffin relation.

FIG. 4 shows total ($\Delta\varepsilon_{tot}$) and inelastic ($\Delta\varepsilon_{inelastic}$) strain ranges vs. the number of cycles to failure (N_f) for F82H-mod in the unirradiated condition and after neutron irradiation at 330-

337 °C to 31 dpa in ARBOR 1 [7] and to 47 dpa in ARBOR 2. The neutron irradiation of F82H-mod to 47 dpa leads to the increase of the lifetime in comparison with unirradiated state for adequate total strain amplitudes. For adequate inelastic strain amplitudes below 0.43 % the lifetime remains nearly unchanged compared to the reference unirradiated state. The apparent increase of the lifetime of the irradiated specimens below $\Delta\varepsilon_{tot} \leq 0.9$ % can be thus mainly related to the irradiation induced material hardening. For adequate inelastic amplitudes above 0.57 %, however, the irradiated specimens exhibit enhanced lifetime compared to the unirradiated state which can not be explained by irradiation hardening only.

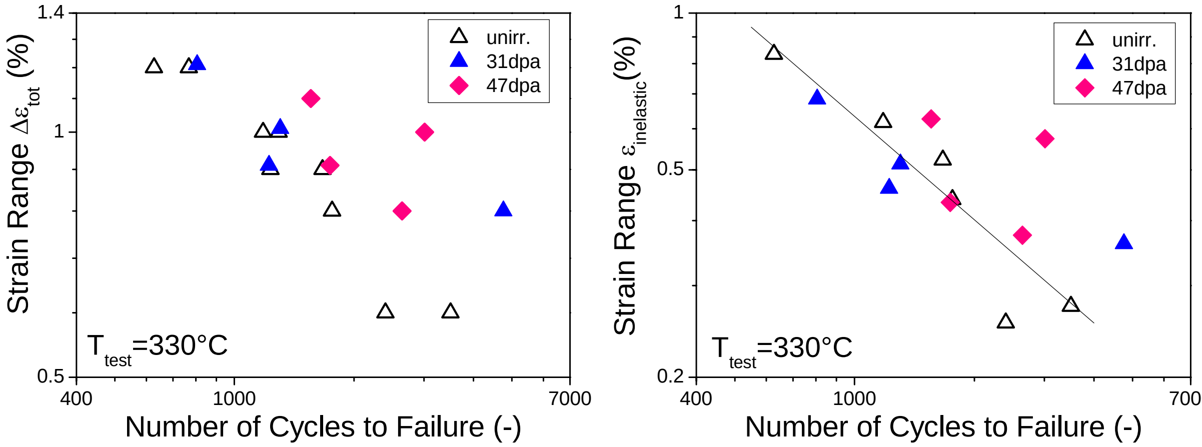
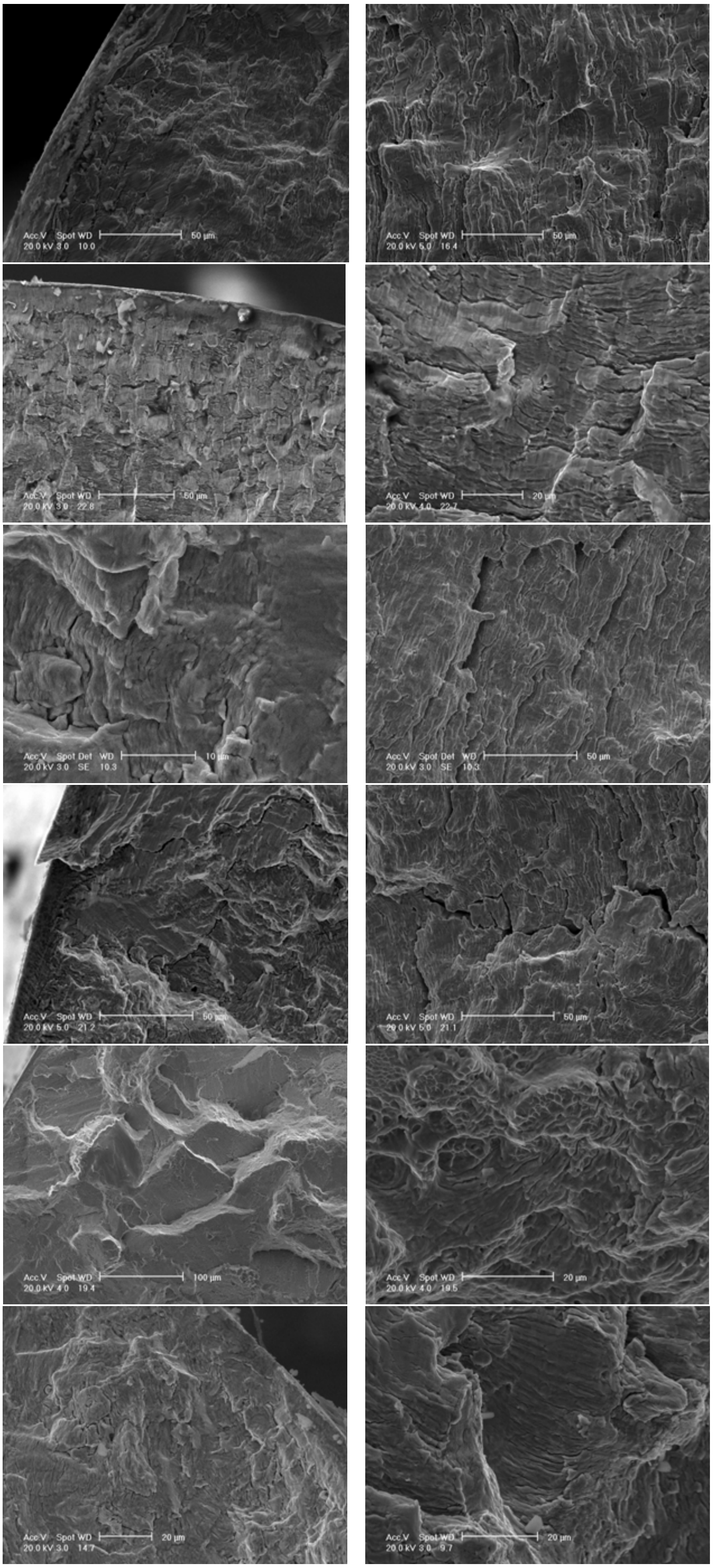


FIG. 4. Fatigue lifetime for unirradiated and up to 71 dpa irradiated (T_{irr} = 330-337 °C) F82H-mod vs. total strain range (left) and inelastic strain range (right); The line represents the description of the unirradiated data by a Manson-Coffin relation.

SEM micrographs of selected RAFM specimens LCF tested after irradiation up to 71 dpa in ARBOR 1 and ARBOR 2 are shown *FIG. 5*. LCF test conditions are indicated in the figure legend. Fractographic investigations of fatigue fracture surfaces indicate appearance of complex, three dimensional fracture surfaces being more pronounced at high damage doses. The fracture surface morphology reveals a terrace-like pattern due to multiple deflections of the fatigue crack along its propagation path. In addition, pronounced emission of the secondary cracks propagating in the axial direction is observable for 71 dpa irradiated specimens. Such a remarkable change of the fatigue crack propagation behaviour seems to have no clear effect on the fatigue lifetime. Indeed, after irradiation to 71 dpa, EUROFER97 and EUROFER97 HT specimens show lifetimes that are comparable to lifetimes for adequate inelastic strain amplitudes in the reference unirradiated states in *FIGs. 1* and *3*. Furthermore for F82H-mod specimens irradiated to 47 dpa, a pronounced coarsening of the fatigue fracture surface morphology for $\Delta\varepsilon_{tot}$= 0.9 % ($\Delta\varepsilon_{inelastic}$=0.43 %) does not affect the lifetime in comparison to the unirradiated state in *FIG. 4*. The specimen tested at $\Delta\varepsilon_{tot}$= 1.0 % ($\Delta\varepsilon_{inelastic}$=0.57 %) in contrast shows the lifetime enhancement in comparison to the unirradiated state.

6. Discussion

The use of the SSTT specimens leads to a considerable underestimation of the fatigue lifetime in the unirradiated condition. The state of the surface finish quality and its possibly different influence on the fatigue lifetime of the SSTT specimens in the unirradiated and irradiated conditions need to be investigated in more details for the unambiguous interpretation of the irradiation influence on the fatigue behaviour.



EUROFER97
E1 21
ARBOR 2
47 dpa/337 °C
1.1 %/330 °C

EUROFER97
E1 10
ARBOR 2
71 dpa/334 °C
1.0 %/330 °C

EUROFER97 HT
E2 01
ARBOR 1
31 dpa/330 °C
1.0 %/330 °C

EUROFER97 HT
E2 12
ARBOR 2
71 dpa/334 °C
0.9 % /330 °C

F82H-mod
F 13
ARBOR 2
47 dpa/ 337 °C
0.9 % /330 °C

F82H-mod
F 15
ARBOR 2
47 dpa/ 337 °C
1.0 % /330 °C

FIG. 5. SEM micrographs of LCF tested selected RAFM specimen. The specimen identifications along with irradiation and LCF test conditions are indicated in figure legend.

In the low cycle fatigue regime the evolution of the fatigue life with inelastic strain can be described by the Manson-Coffin relationship

$$\Delta\varepsilon_{inelastic} = CN_f^m \tag{1}$$

with m and C as material and temperature dependent parameters. The solid lines in *FIGs. 1, 3 and 4* represent the description of the unirradiated data with the above equation. The best fits were obtained with m = -0.68, m = -0.52 and m = -0.66 for EUROFER97, EUROFER97 HT and F82H-mod, respectively. For adequate inelastic strain amplitudes and for the SSTT used in the current work the most of the irradiated data are scattered around the unirradiated data. Considering the large inherent scattering of fatigue lifetime this indicates little or no influence of the neutron irradiation on the fatigue damage evolution. The apparent increase of the fatigue lifetime observed in $\Delta\varepsilon_{tot}(N_f)$ representation in *FIGs. 1,3,4* and in [7] is thus mainly related to the irradiation hardening revealed in monotonic tensile experiments [2]. Comparatively, in [11] the influence of the low temperature neutron irradiation (3.8 dpa at 250 °C) on the fatigue behaviour of F82H was found to be sensitive to the test temperature. The fatigue tests at 250 °C yielded comparable results for the unirradiated and irradiated specimens for adequate inelastic strain amplitudes, whereas a strong reduction of the lifetime in the irradiated condition was found in a RT test at a low total strain range (of 0.4 %) attributed to the occurrence of a channel fracture.

Noticeable reduction of the fatigue lifetime of some EUROFER97 and EUROFER97 HT specimens after irradiation to 47 dpa more pronounced at high (and sometimes intermediate) strain ranges in comparison to the unirradiated state indicates, however, that the inelastic strain amplitude is not the only controlling parameter for the fatigue damage accumulation. The neutron irradiation induced enhanced stress state in comparison to the unirradiated state for adequate inelastic strain amplitudes, as shown in *FIG. 2*, might also accelerate the fatigue damage evolution [10].

Pronounced increase of fatigue lifetime of RAFM steels for low strain ranges ($\Delta\varepsilon_{inelastic} \leq 0.44$ %) after irradiation to 31 dpa in comparison to reference unirradiated state, seen in *FIGs. 1, 3, 5*, is attributed to the strongly reduced fatigue damage evolution at these low strain ranges [10]. An enhancement of lifetime at low (inelastic) strain ranges is, however, no more observable after neutron irradiation to the higher damage doses above 47 dpa in *FIGs. 1, 3, 4.* Irradiation damage remarkably modifies the fracture surface morphology, see *FIG. 5*. Appearance of a complex, three dimensional fracture surfaces, initiation of the secondary cracks propagating into axial direction, considerable coarsening of the fracture surface morphology with increasing the irradiation dose, however, do not show clear effects on the fatigue lifetime.

Conclusion

Isothermal LCF properties of EUROFER97 and F82H-mod steels have been studied after neutron irradiation to displacement damage doses up to 71 dpa at 330-337 °C by using SSTT. The neutron irradiation induced hardening and related suppression of the dislocation mobility may differently affect the fatigue behaviour of the irradiated specimens. The increase of the elastic part of the cyclic deformation and related reduction of the inelastic strain amplitude due to irradiation induced hardening lead to the increase of the fatigue life time especially at low strain ranges. The radiation hardening induced enhancement of the stress state might, however, lead to enhanced damage evolution and hence to lifetime reduction especially at high strain ranges. The limited number of available irradiated specimens does not allow detailed statistical analysis of the LCF results emphasizing a need for further investigations.

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References

[1] N. Baluc *et al.*, Status of R&D activities on materials for fusion power reactors, Nucl. Fusion 47 (2007) S696–S717.
[2] E. Gaganidze, H.-C. Schneider, C. Petersen, J. Aktaa, A. Povstyanko, V. Prokhorov, R. Lindau, E. Materna-Morris, A. Möslang, E. Diegele, R. Lässer, B. van der Schaaf, E. Lucon, Proc. of 22nd IAEA Fusion Energy Conference, 13-18 October 2008 Geneva, Switzerland; Paper FT/P2-1.
[3] E. Gaganidze, H.-C. Schneider, B. Dafferner, J. Aktaa, J. Nucl. Mater. 355 (2006) 83–88.
[4] V. Sh. Sulaberidze et al., Testing Techniques, Facilities and Devices for Irradiation Test Performance in the RIAR Research Reactors, Status and Prospects for Irradiation Experiments in Russia/CIS, Proc. of 32. Japan Workshop, 17 November 2004, Oarai, Japan.
[5] C. Petersen, A. Povstyanko, V. Prokhorov, A. Fedoseev, O. Makarov, B. Dafferner, J. Nucl. Mater. 367–370 (2007) 544-549.
[6] L. R. Greenwood and R. K. Smither, "SPECTER: Neutron Damage Calculations for Materials Irradiations", ANL/FPP/TM-197, 1985.
[7] C. Petersen, A. Povstyanko, V. Prokhorov, A. Fedoseev, O. Makarov, M. Walter, J. Nucl. Mater. 386–388 (2009) 299–302.
[8] J. Rensman, NRG Irradiation Testing: Report on 300 °C and 60 °C Irradiated RAFM Steels, Petten 2005, 20023/05.68497/P.
[9] J. Aktaa, R. Schmitt, Fus. Eng. Des. 81 (2006) 2221–2231
[10] J. Aktaa, C. Petersen, ICFRM 14, Sapporo, Japan, 2009, *accepted for publication* in J. Nucl. Mater. (2010).
[11] Y. Miwa, S. Jitsukawa, M. Yonekawa, J. Nucl. Mater. 329–333 (2004) 1098–1102.