FT/P2-1 Mechanical Properties of Reduced Activation Ferritic/Martensitic Steels after High Dose Neutron Irradiation

Mechanical Properties of Reduced Activation Ferritic/Martensitic Steels after High Dose Neutron Irradiation

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abstract: The Reduced Activation Ferritic/Martensitic (RAFM) steels and their Oxide Dispersion Strengthened (ODS) variants are considered as primary candidate structural materials for fusion reactor breeding blankets with operating temperatures between 250 and 650 °C. The irradiation performance of the European reference steel EUROFER97 was thoroughly studied in various low (IRFUMA up to 2 dpa, SUMO, SIWAS up to 9 dpa), medium (WTZ RUS 01/577, SPICE up to 15 dpa) and high dose (ARBOR 1 up to 33 dpa, ALTAIR up to 42 dpa) irradiation programmes carried out in a wide temperature window from 60 to 450 °C. Although the irradiation damage resistance of EUROFER97 is superior to that of conventional ferritic martensitic steels, the low temperature (<350-400 °C) irradiation hardening, accompanied by embrittlement and reduced ductility, did not reach saturation up to 30 dpa and remains the limiting factor for material application. High dose, up to 70 dpa/330 °C, mechanical properties of RAFM steels were studied in the ARBOR 2 irradiation experiment performed in BOR 60 at SSC RF RIAR. The yield stress and the Ductile-to-Brittle-Transition Temperature (DBTT) of EUROFER97 indicate saturation of low temperature hardening and embrittlement. The evolution of these quantities with irradiation dose can be qualitatively understood within a Whapham and Makin model. The thermal recovery experiments were performed on selected specimens from ARBOR 2. Annealing of irradiated (70 dpa/330 °C) EUROFER97 at 550 °C for 1 h leads to substantial reduction of the yield stress, resulting in a residual hardening of 30 MPa. Annealing at 550 °C for 3 h leads to further reduction of the yield stress and a residual hardening of only 24 MPa is observed. Similar to tensile properties, impact properties are also significantly improved compared to the as-irradiated state in thermal recovery tests. After annealing of 70 dpa irradiated EUROFER97 at 550 °C for 3 h a residual DBTT shift of 48 °C is obtained. Taking into account a need for continuous development and characterisation of materials and welding technologies to be potentially used in ITER-TBM (EUROFER and EUROFER ODS steels) and DEMO (ferritic ODS steels, tungsten alloys) and in order to increase basic scientific knowledge and to develop a materials data base for DEMO design, needs for specific irradiation campaigns are outlined.

1. Introduction

The growing energy demand in the world along with the limited capacity of fossil energy sources makes the development of alternative energy sources indispensable. Fusion research is aimed at demonstrating that this energy source can be used to produce electricity in a safe and environmentally friendly way, with abundant fuel resources. The feasibility of energy generation by means of fusion has to be demonstrated in the Demonstration Power Plant (DEMO) which is intended to be built after the expected successful operation of ITER (International Thermonuclear Experimental Reactor). The development and validation of DEMO relevant structural materials and adequate joining technologies, therefore, belong to the key tasks within the European long term fusion R&D programme.

The Reduced Activation Ferritic/Martensitic (RAFM) steels and their Oxide Dispersion Strengthened (ODS) variants are considered as primary candidate structural materials for breeding blankets (BB) with operating temperatures between 250 and 650 °C [1]. In addition to favourable mechanical and thermal properties, the key demands for the choice of the structural materials are i) resistance to radiation-induced damage phenomena, ii) low neutron induced activation, iii) availability of fusion relevant joining technologies, iv) compatibility with plasma and coolant. With these objectives the European reference 9%Cr-WVTa steel EUROFER97 was specified and industrial batches of 3.5 and 8.0 tons have been produced with a variety of semifinished, quality assured product forms. A large characterization program is being performed including microstructural, mechanical and corrosion experiments.

The irradiation performance of EUROFER97 for the as-delivered (980 °C for 0.5h + 760 °C for 1.5 h) and pre-irradiation heat treated (1040 °C for 0.5 h + 760 °C for 1.5 h) conditions has been thoroughly studied in various *low* (IRFUMA up to 2 dpa [2], SUMO, SIWAS up to 9 dpa [3]), *medium* (WTZ RUS 01/577 [4], SPICE up to 15 dpa [5]) and *high* dose (ARBOR 1 up to 33 dpa [6], ALTAIR up to 42 dpa [7]) European irradiation programmes. The irradiation experiments have been carried out in Material Test Reactors BR2 at SCK-CEN, HFR at JRC and NRG Petten and in BOR 60 at SSC RF RIAR in a wide temperature window from 60 to 450 °C. Although, the irradiation performance of EUROFER97 is superior to that of conventional ferritic/martensitic steels, e.g. MANET [4,5], the low temperature (<350-400 °C) irradiation hardening, accompanied by embrittlement and reduced toughness and ductility, did not reach saturation up to 30 dpa and remains the limiting factor for material application indicating further needs of material development and characterization as well as new approaches in breeding blanket design optimisation.

The present work focuses on the investigation of irradiation induced embrittlement and hardening of the European RAFM reference steel for the first wall (FW) of a DEMO fusion reactor, EUROFER97 and selected RAFM steels after high dose neutron irradiation within the ARBOR 2 irradiation project. For the interpretation of the results the data from the previous *low* and *medium dose* irradiation programmes are also assessed. Special emphasis is put on the investigation of the effects of post irradiation annealing on the recovery of the mechanical properties of RAFM steels.

2. Materials

An industrial batch of the European RAFM steel EUROFER97 (nominal composition Fe-9Cr-1.1W-0.2V-0.12Ta, see e.g. [5] for details) 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/0.5 h 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. 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 (1040 °C/0.5 h + 760 °C/1.5 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 mm, 15 mm and 25 mm plates have been distributed by IEA, and subsequently by FZK to the European partners. For the ARBOR irradiations the specimens have been machined from the 25 mm plate subjected to a heat treatment of 1040 °C/38 min + 750 °C/2 h.

3. Irradiation Experiments

The BOR 60 experimental fast reactor of SSC RF RIAR - nowadays widely used as irradiation facility for material science purposes - offers different irradiation positions in the reactor core of 450 mm height and 550 mm diameter [8]. For the study of the high dose irradiation performance of EUROFER97 and other RAFM steels, 50 % of the specimens from the irradiation experiment ARBOR 1 [6] (**A**ssociated **R**eactor Irradiation in **BOR** 60) were reloaded into the ARBOR 2 rig for further irradiation to reach a target damage 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 values for ferritic steel specimens was conducted using the SPECTER code [9]. A cumulative damage dose up to 70 dpa has been achieved in the fast neutron spectrum of the BOR 60 reactor, i.e. (> 0.1 MeV) of 1.8×10^{15} n/cm².

The ARBOR 2 irradiation included 90 mini-tensile/Low Cycle Fatigue (LCF) specimens and 60 mini-impact (KLST) specimens of 9 different RAFM steels. KLST specimens were machined parallel to the rolling direction of the material plates (L–T orientation), see e.g. [5] for specimen geometry. Small size cylindrical specimens of 7.6 mm gauge length and 2 mm diameter were used for the investigation of tensile and LCF properties [4].

4. Post Irradiation Mechanical Testing

The post irradiation mechanical testing of the specimens from the ARBOR 2 irradiation is performed at the material science laboratory of SSC RF RIAR under ISTC Contract Nr. # 2781p.

Tensile tests are performed with an electro-mechanical testing machine INSTRON 1362 DOLI, equipped with a three-zone furnace and high-temperature MAYTEC extensometer [6]. Specimens are tested under static (tensile) loading at different temperatures (250, 300 and 350 °C) with a strain rate of 3×10^{-3} s^{-1}. From the load-displacement curves, strength and strain quantities like the 0.2% offset yield stress ($R_{p0.2}$), ultimate tensile strength (R_m), uniform strain (A_g) and total strain (A) are calculated. Reduction of area (Z) was measured from photos of the broken specimens taken after testing.

Impact tests are performed with a modern ZWICK 5113-HKE instrumented impact testing facility, equipped with a pendulum hammer of 25 J impact energy [6]. The test and evaluation procedures are identical to those employed in previous investigations, see e.g. [6]. The impact energies (E) vs. test temperature (T) curves were analyzed with respect to the upper shelf energy (*USE*) and the ductile-to-brittle transition temperature (*DBTT*) as described in [5].

5. Experimental Results

FIG. 1 shows yield stress ($R_{p0.2}$) vs. test temperature (T_{test}) for EUROFER97 in the unirradiated condition and after neutron irradiation in different *medium* and *high* dose European irradiation programmes at *target* irradiation temperatures (T_{irr}) between 300 and 350 °C.

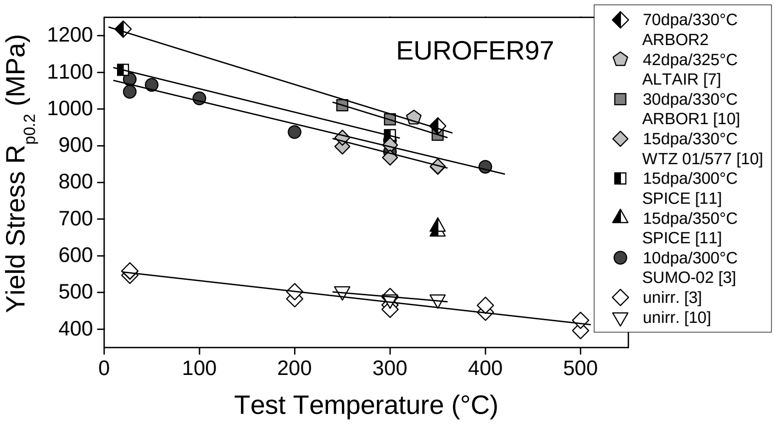

FIG. 1. Yield stress $R_{p0.2}$ vs. test temperature for EUROFER97 in the unirradiated condition and after neutron irradiations in different European irradiation programmes (irradiation conditions and programmes are given in the figure legend). The lines are a guide for the eye.

Neutron irradiation leads to substantial increases in the yield stress which is sensitive to the irradiation parameters *i.e.* irradiation dose and temperature. Furthermore, for given irradiation conditions the yield stress increase depends on the test temperature and is larger at low test temperatures. The specimens from ARBOR 2 show the highest yield stress at test temperatures of 20 and 350 °C. While the differences in the yield stress values at 300 and 330 °C irradiations up to 15 dpa are still moderate and within data scatter, as can be seen from the comparison between WTZ [10] and SPICE [11] results, yield stress values after 15 dpa/350 °C irradiation are considerably lower indicating substantial thermal recovery at this irradiation temperature.

FIG. 2 presents the evolution of the irradiation hardening with dose for irradiation temperatures between 300 and 330 °C and for a test temperature of 300 °C. An exception is the data point from ARBOR 2 experiment which was obtained at T_{test} = 350 °C. This can be an explanation for the observed lower hardening in comparison to ARBOR 1 [10] and ALTAIR [7] results. In spite of large data scattering partly due to different irradiation temperatures a clear tendency to saturation at the achieved irradiation doses is identified.

FIG. 3 shows the impact energy vs. test temperature curves for EUROFER97 for the unirradiated condition and after neutron irradiation in different European campaigns at *target* irradiation temperatures between 300 and 350 °C. The neutron irradiation below 330 °C strongly degrades the impact properties leading to the shift of the *DBTT* towards higher temperatures and reduction of the *USE*. At the achieved doses a clear tendency to saturation of impact properties is identified. For 69.8 dpa/335 °C irradiation the *DBTT* is found to be 152 °C yielding an irradiation-induced shift in *DBTT* (Δ*DBTT*) of 233 °C. This value is only slightly higher than the irradiation-

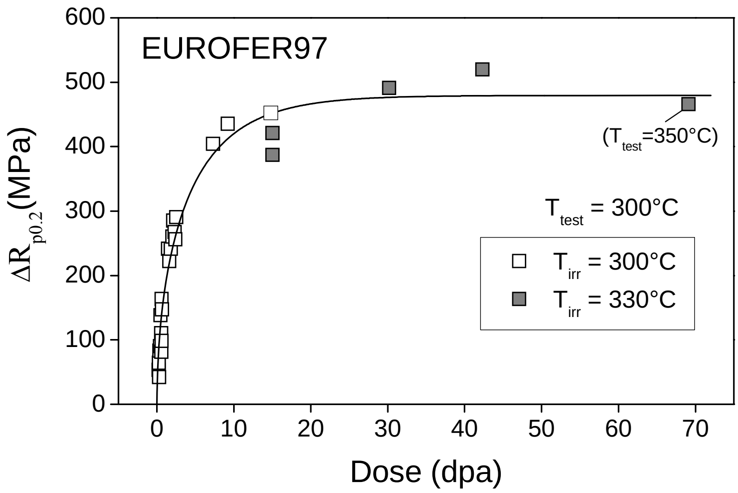

FIG. 2. Irradiation hardening vs. irradiation dose for T_{irr}=300-330 °C and T_{test}=300 °C (exception: specimen from ARBOR 2 is tested at T_{test}=350 °C) of EUROFER97. For comparative purposes the low dose data from [2,3] are also included. The line is a model description of the entire data set.

induced shift in the *DBTT* after 32 dpa/ 332 °C irradiation (Δ*DBTT* =218 °C). Similarly, the *USE* of 6.6 J of ARBOR 2 irradiated EUROFER97 is only slightly lower than that of the ARBOR 1 irradiation (7.0 J). The SPICE neutron irradiation at 350 °C leads to minor embrittlement of the steel. Indeed, the *DBTT* for the 17.4 dpa/ 350 °C irradiation lies well below RT.

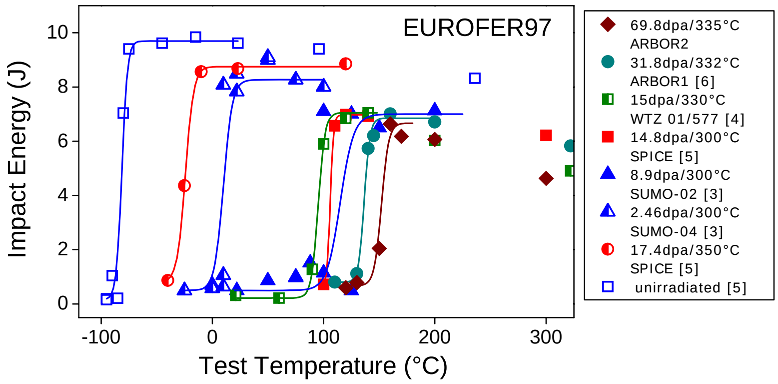

FIG. 3. Impact energy vs. test temperature of EUROFER97 in unirradiated condition and after neutron irradiations in different European irradiation programmes (irradiation conditions and programmes are given in the figure legend). The specimens are machined from the as-delivered 25 mm plate. The lines are fits to the ductile-to-brittle-transition regions as described in [5].

FIG. 4 shows the evolution of the irradiation induced embrittlement with irradiation dose for EUROFER97 and F82H steels at irradiation temperatures between 300 and 330 °C. For EUROFER97 a differentiation is made between specimens machined from as-delivered products and specimens machined from the plates subjected to pre-irradiation heat treatment. The results on F82H and F82H-mod are plotted together for different heat treatments and for different material compositions [12]. These circumstances partly explain the large data scatter observed for F82H. The pre-irradiation heat treatment of EUROFER leads to considerable improvement of the irradiation resistance at damage doses up to 30 dpa.

The results of the post-irradiation annealing experiments on the EUROFER specimens from the WTZ 01/577 and ARBOR 2 irradiations are presented in *FIGS. 5* and *6*. *FIG. 5* shows yield stress and ultimate tensile strength for EUROFER97 in the as-irradiated condition and after post-irradiation annealing at 550 °C and for 1-3 h, normalized to the unirradiated values. Annealing of irradiated (70 dpa/ 332 °C) EUROFER97 at 550 °C already for 1 h leads to substantial reduction of the yield stress, resulting in a residual hardening of 30 MPa. Annealing at 550 °C for 3 h leads to further reduction of the yield stress and a residual hardening of only 24 MPa is obtained. Similarly, post irradiation annealing leads to almost complete recovery of the ultimate tensile strength.

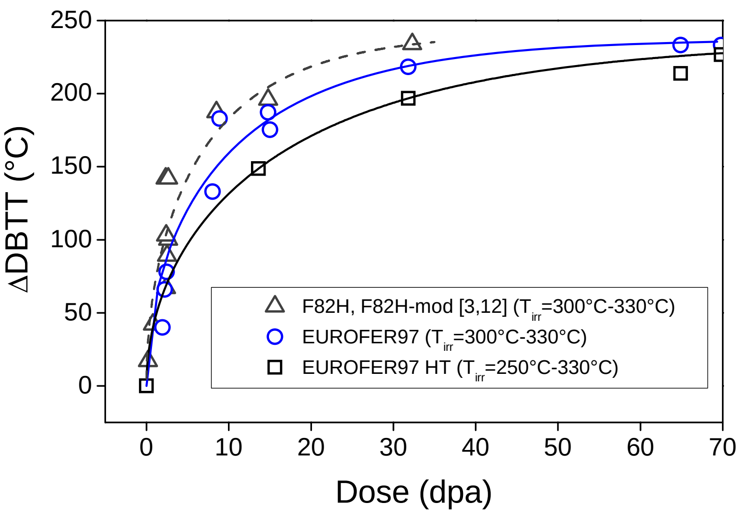

FIG. 4. Irradiation shifts of the DBTT vs. irradiation dose for EUROFER97 and F82H steels. The irradiation temperatures are indicated in the figure legend. The solid lines are a model description of the data. The dashed line is a guide for the eye.

FIG. 6 shows impact energy vs. test temperature curves

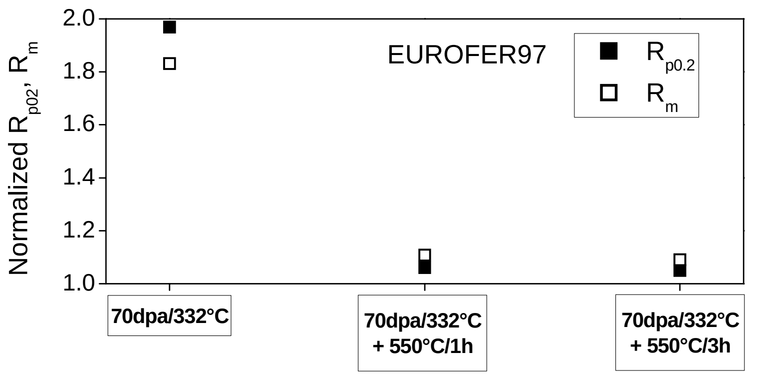

FIG. 5. Yield Stress and ultimate tensile strength for irradiated EUROFER97 (ARBOR 2) normalized with respect to the corresponding unirradiated values in the as-irradiated condition and after post-irradiation annealing.

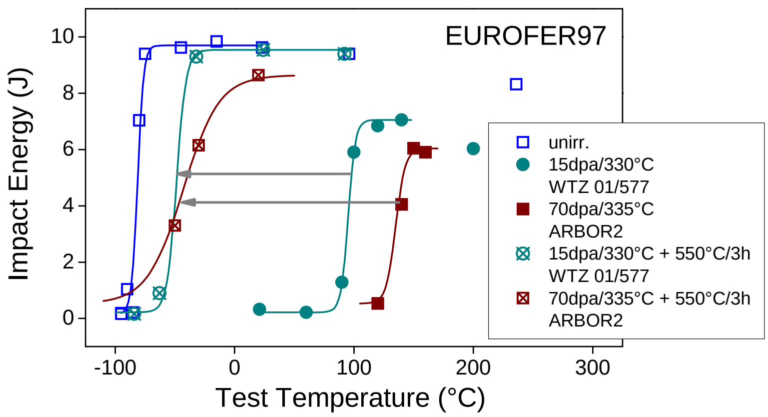

FIG. 6. Impact energy vs. test temperature for EUROFER from the WTZ 01/577 and ARBOR 2 irradiations in the as irradiated condition and after post-irradiation annealing at 550 °C for 3 h. The results in the reference unirradiated condition are also included. The lines are fits to the ductile-to-brittle-transition regions as described in [5]. The arrows indicate recovery of the DBTT.

for EUROFER specimens from the WTZ 01/577 and ARBOR 2 irradiations in the as-irradiated condition and after post-irradiation annealing at 550 °C and for 3 h. Similar to tensile properties, impact properties are also substantially improved in thermal recovery tests compared to the as-irradiated state. The post-irradiation annealing of e.g. 70 dpa irradiated EUROFER97 HT at 550 °C for 3 h led to a reduction of the *DBTT* from 135 to -43 °C resulting to a residual embrittlement of just Δ*DBTT* = 48 °C.

6. Discussion

There is a close correlation between neutron irradiation induced hardening and embrittlement of RAFM steels at irradiation temperatures below 350 °C. The mechanical properties, however, are mostly influenced at irradiation temperatures T_{irr} ≤300-330 °C, see also [5,13].

Irradiation-induced material hardening can be described by the Whapham and Makin model [14], according to the following relationship:

$$\Delta\sigma = \Delta\sigma_s \sqrt{1 - \exp\left(-\frac{\Phi}{\Phi_o}\right)} \qquad (1)$$

where $\Delta\sigma_s$ is a saturation value of hardening, Φ the irradiation dose and Φ_o a scaling dose characterizing how fast the saturation in hardening sets in. The line in *FIG. 2* is a description of the irradiation induced hardening according to Eq. (1) with $\Delta\sigma_s$=480 MPa and Φ_o=6.84 dpa. In spite of (i) differences in the irradiation conditions e.g. the irradiation temperature, the neutron flux density, (ii) differences in test conditions e.g. specimen geometry, strain rate and (iii) scatter of experimental data, Eq. (1) describes qualitatively the evolution of hardening with irradiation dose. The hardening rate, furthermore, appears to be significantly decreased at the achieved damage doses. Recently, the evolution of irradiation induced defects with irradiation dose underlying Eq. (1) was successfully incorporated in a coupled viscoplastic deformation damage model in [15]. The model not only did account for the irradiation induced hardening of EUROFER but also yielded a successful description of alteration of hardening under inelastic deformation and high temperature dwell conditions.

The neutron-irradiation induced embrittlement for EUROFER97 has reached saturation at achieved damage doses in *FIG. 4*. Due to the close correlation between low temperature hardening and embrittlement, the evolution of embrittlement with irradiation dose can be qualitatively described by an equation of the type $\Delta DBTT = \Delta DBTT_s(1-\exp(-\Phi/\Phi_o))^{1/2}$ with $\Delta DBTT_s$ as the saturation value of the embrittlement. The solid lines in *FIG. 4* are fits to the data with the above equation. The results for EUROFER97 are best described with $\Delta DBTT_s$ =238 °C and Φ_o=16.7 dpa. Remarkably, for EUROFER97, Φ_o determined for *tensile* hardening (in *FIG. 2*) is substantially less than Φ_o determined for *impact* embrittlement (in *FIG. 4*), indicating that *tensile* hardening saturation is reached at lower levels of damage dose. This observation would suggest the existence of a non-hardening *impact* embrittlement mechanism in addition to hardening embrittlement, the former becoming dominating at high irradiation doses. It must be born in mind that the impact deformation occurs at orders of magnitude higher rate affecting the mechanism in operation.

Post-irradiation annealing led to substantial improvements of the mechanical properties of EUROFER97 indicating recovery of the radiation induced damage. There is a nearly complete recovery in the tensile properties. The recovery in the impact properties, however, seems to be sensitive to prior irradiation damage. Indeed, though the *DBTTs* are similar after post-irradiation annealing of WTZ and ARBOR 2 specimens, the transition region seems to be broadened for ARBOR 2 specimens. The repeatability, of the mechanical properties recovery under repeatable irradiation and annealing conditions is one of the critical issues for validation of this method for application to the FW and BB structures of a future fusion reactor.

7. Summary and Outlook

Neutron irradiation leads to severe degradation of the mechanical properties of the RAFM steels at low irradiation temperatures below T_{irr} ≤300-330 °C. Neutron irradiation-induced hardening and embrittlement indicate saturating behaviour at the achieved damage doses for these low irradiation temperatures. The evolution of hardening and embrittlement with irradiation dose can be qualitatively described using Whapham and Makin's model. Ongoing and planned quantitative microstructural investigations are mandatory to get deeper insight in the radiation damage mechanisms and for a quantitative description of the neutron irradiation induced hardening and embrittlement within appropriate models.

The state-of-the-art structural materials are highly suited for the special fusion reactor design with the operating temperature range for the FW and BB being between 350 and 450 °C. The thermal recovery experiments yielded very promising results. After possible validation of this method through the study of the repeatability of these experiments, recovery heat-treatments can also be utilized for extension of the operating temperature range down to RT.

There is a need for continuous development and characterisation of materials. First priority on a 5-10 years schedule is the understanding and characterization of materials used with ITER-TBM, i.e. EUROFER base material and welding technologies. EUROFER ODS steels are being developed and characterized with a potential use for TBM, too. Within a 10-20 years frame, novel divertor materials like ferritic ODS steels and improved tungsten alloys with suitable properties should be developed together with adequate joining technologies. In order to increase the basic scientific knowledge and to develop a materials data base for DEMO design, several specific irradiation campaigns should be launched (a) at relevant neutron doses (more than 10 dpa), (b) at appropriate temperatures (250-650 °C), (c) addressing the most critical mechanical properties and- finally, (d) coupled with comprehensive microstructural investigations.

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