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Impact property degradation of ferritic/martensitic steels after the fast reactor irradiation 'ARBOR 1'

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Abstract

In an energy generating fusion reactor, structural materials will be exposed to very high levels of irradiation damage of about 100 dpa. These damage conditions can be realized – in reasonable times – only in fast reactors. For this purpose a cooperation between Forschungszentrum Karlsruhe and State Scientific Centre of Russian Federation Research Institute of Atomic Reactors had been implemented. The irradiation project is named 'ARBOR 1' (Latin for tree). Impact, tensile and low cycle fatigue specimens of reduced activation ferritic/martensitic steels, e.g. EUROFER 97, F82H mod., OPTI-FER IVc, EUROFER 97 with different boron contents and ODS-EUROFER 97 have been irradiated in a fast neutron flux of 1.8×10^{15} n/cm² s (>0.1 MeV) at a temperature <340 °C up to ~30 dpa. In the post irradiation impact tests a dramatic increase in the ductile to brittle transition temperature as an effect of irradiation has been detected. © 2007 Elsevier B.V. All rights reserved.

1. Introduction

In an energy generating fusion reactor, structural materials will be exposed to very high levels of irradiation damage of about 100 dpa. A simulation facility – like IFMIF – is not available in the nearer future to study the materials behavior under fusion relevant irradiation conditions, e.g. specific He/dparatio. Therefore it has been planned to introduce displacement damages to high levels in fast reactors.

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Due to the fact that fast reactor irradiation facilities in Europe are no longer available, a cooperation between Forschungszentrum Karlsruhe (FZK) and State Scientific Centre of Russian Federation Research Institute of Atomic Reactors (SSC RF RIAR) has been implemented. The irradiation project 'Associated Reactor Irradiation in BOR 60' is named 'ARBOR 1' (Latin for tree).

The BOR 60 experimental fast reactor of SSC RF RIAR is nowadays widely used as an irradiation facility for material science purposes. With a reactor core dimension of 450 mm height and 550 mm in diameter, different irradiation positions are available. In the selected cell D-23, direct temperature measurement by thermocouple during irradiation is possible [1].

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The ARBOR 1 irradiation device with outer hexagon size of 45 mm and a specimen capsule diameter of 39 mm is heated by the direct flow of the sodium coolant from the reactor high-pressure chamber, which allows a coolant flow rate of 7 m³/h and a low gamma heating rate of 5 W/g, which produces an increase of about 10–15 °C over the length of the capsule.

The irradiation rig was instrumented; neutron monitors in the central tube and three temperature detectors at three of ten levels of specimen positions were equipped. The calculation of the damage dose values for ferritic steel specimens was conducted using the SPECTER code [2]. In this case a neutron energy spectrum in cell D-23 was used that had been measured in previous dosimetry experiments.

The ARBOR 1 irradiation included 150 mini-tensile/low cycle fatigue specimens and 150 mini-impact (KLST) specimens of nine different RAFM steels, e.g. EUROFER 97, F82H mod., OPTIFER IVc, EUROFER 97 with different boron contents and ODS-EUROFER 97. They were irradiated in a fast neutron flux of 1.8×10^{15} n/cm² s (>0.1 MeV) at a temperature <340 °C up to ~30 dpa [3]. Approximately 50% of the specimens were unloaded for post irradiation examinations (PIE). The other 50% were reloaded into the ARBOR 2 rig for further irradiation in BOR 60 to reach a maximum irradiation damage of 70 dpa.

The mechanical testing PIE is performed at a material science laboratory of the SSC RF RIAR. The impact testing results reported here are the first part of the PIE that also includes tensile and fatigue testing. Since tensile test results are still incomplete and low cycle fatigue tests will start in December 2005, we concentrate in this paper on impact data. The impact tests are performed with a modern instrumented impact testing facility of Zwick 5113-HKE type, equipped with a pendulum hammer of 15 J impact energy and installed in the VK-39 hot cell of the SSC RF RIAR.

2. Experimental details

Small size KLST specimen with the dimensions $27 \times 4 \times 3$ mm were utilized as L–T specimens, i.e. axis into rolling direction and flaw into long transverse direction, to investigate the impact properties after irradiation of the following materials, as previously reported with their chemical composition [3]. The European reduced activation ferritic/martens-

itic (RAF/M) steel EUROFER 97 is included in two annealing conditions. EUROF 1: EUROFER 97, heat E83697, (as received: 980 °C 31 min/air $cooled + 760 \circ C$ 90 min/air cooled), EUROF 2: EUROFER 97, heat E83697, (1040 °C 31 min/air $cooled + 760 \circ C 90 min/air cooled$). EUROF 1 is optimised for good tensile and fatigue resistance and EUROF 2 for good Charpy ductile to brittle behavior. The Japanese RAF/M steel F82H mod. is included as the international reference steel: F82H mod., heat 9741, (as received: 1040 °C $38 \text{ min/air cooled} + 750^{\circ}\text{C} 2 \text{ h/air cooled}$). The German developed OPTIFER IVc, OPT IVc, heat 986779, (950 °C 30 min/air cooled + 750°C 2 h/air cooled), is included as reference material for comparison to data from the HFR-irradiation [4]. The three materials ADS 2, ADS 3 and ADS 4, based on EUROFER 97, are experimental heats to study the influence of He on RAF/M-steels. ADS 2 is an EUROFER 97-steel with 82 wppm natural boron (B), heat 806, (1040 °C 31 min/air cooled + 760°C 90 min/air cooled), ADS 3 an EUROFER 97-steel with 83 wppm 10 B, heat 826, (1040 °C 31 min/air cooled + 760 °C 90 min/air cooled) and ADS 4 an EUROFER 97-steel with 1160 wppm ¹⁰B, heat 825, (1040 °C 31 min/air cooled + 760°C 90 min/ air cooled).

An important feature of this ARBOR 1 irradiation was the inclusion of specimens of mechanically alloyed EUROFER 97 with 0.5% Y₂O₃, the recently developed higher creep-rupture resistant RAF/ M-steels. The specimen designation is EUROD-Ship, heat HXN 958/3, (as received: 980 °C 31 min/air cooled + 760 °C 90 min/air cooled).

The procedure for testing and evaluation was the same as in the previous investigations of ferritic-martensitic steels which were carried out after irradiation to lower damage doses [4].

The main test features are: a pendulum hammer with 15 J impact energy, a tup of 2 mm radius, span of 22 mm, a PC-controlled testing and data acquisition/processing procedure, automatic control of the feeding system to minimize the specimen temperature distribution.

The primary 'force vs. time' curves are used to determine the impact energy for each specimen. These values are plotted vs. testing temperature and the ductile to brittle transition temperature (DBTT) is derived as the temperature at which the energy corresponds to half upper shelf energy (USE/2) that is one of the characteristic values used to define the DBTT.

3. Results and discussion

Results for the technically relevant materials EUROFER 97. F82H mod. and for comparison OPTIFER IVc are plotted in the graphs below. The OPTIFER IVc can be compared with OPTI-FER Ia from FZK's 300 °C lower dose irradiation experiment MANITU [4]. Since we implemented in the ARBOR-1 irradiation only 25 mm plate material of EUROFER 97 and F82H mod., it is possible to compare those data with results from the NRG 300 °C lower dose irradiation experiments, e.g. SUMO-02, SUMO-04 and CHARIOT-04. In case of the FZK's EURODShip, which is a powder hipped EUROFER 97 with 0.5% Y₂O₃, a comparison can be made with the EUROFER 97 powder HIP ODS from CEA, irradiated in the SUMO-07 irradiation [5].

In Fig. 1 the impact curves for EUROF 1 and EUROF 2 are shown for the unirradiated condition and for material irradiated at 332 °C to 31.8 dpa as an average value. The transition temperatures (DBTT) for unirradiated materials lie very close together (EUROF 1: -81 °C and EUROF 2: -90 °C), the USE-values for both materials are 9.84 J. For comparison, the DBTT and USE of NRG impact tests had been found at -68 °C and 9.5 J, respectively. The DBTT for 31.8 dpa irradiated EUROF 1 is 137 °C (NRG-DBTT-data: 10 °C (SUMO-04, 300 °C, 2.46 dpa) and 115 °C (SUMO-02, 300 °C, 8.9 dpa)) and for EUROF 2 is 107 °C.

The irradiation induced shift in DBTT (ΔDBTT) for EUROF 1 is 218 °C and for EUROF 2 is 197 °C. The USE is reduced in the irradiated state, to 7.01 J for EUROF 1 (NRG-USE-data: 8.5 J (SUMO-04, 300 °C, 2.46 dpa) and 7.07 J (SUMO-02, 300 °C, 8.9 dpa)) and 6.76 J for EUROF 2. So, on one hand the earlier finding [4] was confirmed that the higher austenizing temperature of EUROF 2 at 1040 °C reduces the irradiation damage in respect to impact properties, mainly on DBTT. On the other hand the DBTT increases with increasing irradiation damage and a saturation value, as it was expected for RAF/M steels in [6] has not been reached at the irradiation damage of around 30 dpa.

Fig. 2 gives the impact curves for F82H mod. in the unirradiated condition and irradiated at 332 °C to 32.3 dpa (average value). The DBTT for the unirradiated condition was -72 °C, the USE-value 9.41 J. The DBTT and USE of NRG impact tests had been found at $-85 \,^{\circ}$ C and 9.57 J, respectively. The DBTT for 32.3 dpa irradiated F82H mod. is 148 °C (NRG-DBTT-data: 10 °C (CHARIOT-04, 300 °C, 2.64 dpa) and 110 °C (SUMO-02, 300 °C, 8.5 dpa)). The irradiation induced shift in DBTT (Δ DBTT) is thus for F82H mod. 220 °C. The USE is reduced in the irradiated state to 5.03 J for F82H mod. (NRG-USE-data: 9.0 J (CHARIOT-04, 300 °C, 2.64 dpa) and 6.0 J (SUMO-02, 300 °C, 8.5 dpa)). This reduction in USE after irradiation is much higher for F82H mod. than for EUROFER 97 in both modifications. Also, FZK's MANITU



Fig. 1. KLST impact transition curves for unirradiated and irradiated EUROFER 97 of 25 mm plate for two heat treatment conditions.



Fig. 2. KLST impact transition curves for unirradiated and irradiated F82H mod. of 25 mm plate for as received condition.

irradiation [4] has provided impact data on irradiated F82H mod.: DBTT and USE of FZK's unirradiated F82H mod. were -70 °C and 10.7 J, respectively. Irradiated DBTT-data: -23 °C, Δ DBTT-data: 47 °C and USE-data: 10.4 J (MAN-ITU, 300 °C, 0.8 dpa).

The impact behaviour of OPTIFER IVc is shown in Fig. 3. The general tendency is very similar to the above reported materials, but OPTIFER IVc shows the lowest DBTT of 48 °C after the ARBOR-1 irradiation conditions of 332 °C and 32.3 dpa. The reduction of USE after irradiation is very similar to F82H mod. The impact values of unirradiated and irradiated boron doped EUROFER 97 is given in Table 1 and will not be discussed in detail, because their DBTT-values after irradiation are not relevant to practical use of the material in fusion technology. These specimens will be examined microstructurally to study the effect of helium on the mechanical behaviour after irradiation. One



Fig. 3. KLST impact transition curves for unirradiated and irradiated OPTIFER IVc of 25 mm rectangular bar.

Table 1	
Results of impact tests on KLST specimens from the ARBOR 1 irradiation experiment	

Materials, irradiation conditions	DBTT irradiation (°C)	DBTT irradiation (°C)	ΔDBTT (°C)	USE unirradiation (J)	USE irradiation (J)	ΔUSE (J)
EUROF 1, 332 °C, 31.8 dpa	-81	137	218	9.84	7.01	-2.83
EUROF 2, 332 °C, 31.8 dpa	-90	107	197	9.84	6.76	-3.08
F82H mod., 332 °C, 32.3 dpa	-72	148	220	9.41	5.03	-4.38
OPT IVc, 332 °C, 32.3 dpa	-105	48	153	9.12	5.84	-3.28
ADS $2 = EUROF 1 + 82$ wppm natural B 332 °C, 22.4 dpa	-74	174	248	8.81	5.60	-3.21
ADS $3 = EUROF 1 +$ 83 wppm ¹⁰ B, 332 °C, 22.4 dpa	-100	174	274	8.92	5.78	-3.14
ADS $4 = EUROF 1 +$ 1160 wppm ¹⁰ B, 332 °C, 32.3 dpa	-12	260	272	5.50	0.67	-4.83
EURODShip = EUROF 1 + 0.5% Y ₂ O ₃ , 332 °C, 31.8 dpa	135	382	247	2.54	1.51	-1.03

can note that maximum values of $\Delta DBTT$ (248 K, 274 K and 272 K) were observed for boron doped steels.

Also reported in Table 1 are first results of FZK's ODS-EUROFER 97 with 0.5% Y₂O₃. However, since the material was not optimized in respect to thermal treatment, the unirradiated reference impact values with a DBTT of 135 °C and an USE of 2.54 J did not meet any technical application criteria. But the potential is high to decrease DBTT and increase USE as NRG-CEA results

demonstrate [5,7]. Also CEA ODS-EUROFER 97 with 0.5% Y_2O_3 and FZK's thermally treated ODS-EUROFER 97 with 0.3% Y_2O_3 show DBTT-values of -5 °C and -20 °C and USE-values of 3.9 J and 7.0 J, respectively, in the unirradiated state. The CEA ODS-EUROFER 97 with 0.5% Y_2O_3 tested after the SUMO-07 irradiation of NRG (300 °C, 3.1 dpa) exhibited a DBTT of 100 °C and a USE of 2.3 J.

The state of knowledge on the impact behaviour of RAF/M steels is illustrated in Fig. 4. This figure



Fig. 4. Comparison of irradiation dependence on the Ductile to Brittle Transition Temperature behavior for different technically relevant RAF/M steels compared to conventional 12% Cr steel MANET-I.

shows the irradiation damage dependence of DBTT of EUROFER 97, F82H mod. and OPTIFER's, and compares them to the conventional steel MANET-I. The increase in DBTT with increasing irradiation damage in the RAF/M's is actually half of that of MANET-I. However the hope to have reached a saturation state is not confirmed. Further

information will come from the joint ARBOR 2 irradiation with specimens from FZK and CEA irradiated to a damage of 70–80 dpa, respectively, [8].

4. Conclusions

The principle result of the impact tests is a considerable shift in DBTT to higher temperatures for all materials after damage doses of about 30 dpa at 330 $^{\circ}$ C in the ARBOR 1 irradiation.

Generally, and particularly in respect to the ODS modification of EUROFER 97, the data lead to the conclusion that these materials are still retaining technically usable properties.

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