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Eurofer Steel, Development to Full Code Qualification

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Abstract

European Union fusion materials community has been developing a reduced activation martensitic steel called Eurofer for almost 2 decades. This steel has now reached maturity and is being proposed for addition to RCC-MRx. At first, its code qualification is sought for ITER Test Blanket Modules, where the maximum irradiation dose is low, <3 dpa, reactor pulses are of short durations, <500 s, and nominal service temperature does not exceed 550°C. The ultimate goal, however, is to extend its qualification to DEMO and fusion power reactors, where irradiation doses >70 dpa and continuous operation are anticipated. In this paper, after a brief recall of the Eurofer steel development, types of materials and joints used during its characterization are presented. This is followed by the procedures employed for establishment of its materials properties databases and determination of its design allowable values, with emphasis on creep, fatigue and creep-fatigue properties.

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Keywords: Fusion, Eurofer, RCC-MRx, Creep, Fatigue

1. Introduction

European Union fusion materials community has been developing a reduced activation martensitic steel, Eurofer, for application in DEMO and fusion power reactors for almost 2 decades [1,2]. This steel has now reached maturity and is ready for code qualification [3,4]. For this purpose, Eurofer materials properties data have been collected [5-19], analyzed and after validation entered in relational databases allowing full tractability of each datum point back to product information and laboratory test files. These databases have been then used to write the materials procurements files and the materials support documents and design allowable tables following the RCC-MRx procedures.

Initially, the code qualification is sought for application in the 2 EU Test Blanket Modules [20-22]: Helium Cooled Lithium Lead (HCLL) and Helium Cooled Pebble Bed (HCPB). In these modules the maximum irradiation dose will be less than about 3 dpa and service temperatures ≤ 550°C. The ultimate goal, however, is to extend the qualification to DEMO and power reactors where continuous operation and irradiation doses >70 dpa with high helium concentrations are expected [23]. In anticipation of possible changes to Eurofer

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specifications for DEMO or power reactors, the present material is designated Eurofer-TBM, covering the 3 main commercial heats produced so far: Eurofer97-1, Eurofer97-2 and Eurofer97-3.

The French RCC-MRx code is chosen for Eurofer. This is a new reactor construction design code that combines and replaces former RCC-MR (fast breeder) and RCC-Mx (Jules-Horowitz reactor) codes. RCC-MRx will include, in addition to the usual sections on physical properties, borderline properties, analysis data 1 (tensile, fatigue, fracture and fatigue crack propagation properties), analysis data 2 (thermal ageing, creep and creep-fatigue), a new section, analysis data 3, where materials properties are evaluated in the irradiated state.

2. Materials

Several large industrial heats of the Eurofer steel (Eurofer97-1, Eurofer97-2, Eurofer97-3) have been procured to the specification given in Table 1. Samples of these products (mainly bars and plates) have been dispatched to European laboratories† for examinations and characterizations [1]. All the laboratories have tested the materials in the as-received condition (e.g. plates annealed for about 30 minutes at 980°C followed by cooling in air and temper at 760°C for 90 min) and welds in tempered condition (post-weld annealed at 750-760°C for 2 h). Some laboratories have also investigated tube and bar products, effects of additional heat treatments, as well as, different joining technics.

The only discrepancy observed amongst different heats and products examined and tested is lower tensile yield strength for 100 mm bars of Eurofer-2 in comparison with plates.

Table 1. Specified compositions for Eurofer97 in wt.%, unless otherwise stated. (Target values are indicated in [ ]).

Elements	Eurofer97 Steel
Cr	8.5 – 9.5 [9.0]
C	0.09 – 0.12 [0.11]
Mn	0.20 – 0.60 [0.40]
P	< 0.005
S	< 0.005
V	0.15 – 0.25
B	< 0.001
N2	0.015 – 0.045 [0.030]
O2	< 0,01
W	1.0 - 1.2 [1.1]
Ta	0.06 - 0.09
Ti	< 0.01 (100 ppm)
Nb	[<10 ppm]
Mo	[< 50 ppm]
Ni	[< 50 ppm]
Cu	[< 50 ppm]
Al	[<100 ppm]
Si	< 500 ppm
Co	[< 50 ppm]
Sn	As+Sn+Sb+Zr < 100 ppm
As	As+Sn+Sb+Zr < 100 ppm

† CEA/France, KIT/Germany, NRG/ Netherlands, ENEA/ Italy, CIEMAT/ Spain, EPFL/ Switzerland, SCK/ Belgium, Risö/ Denmark, Demokritos/ Greece

3. Database

Materials properties data collected [5-19], have been filtered and only those meeting code qualification standards retained and added to the database. For instance, fatigue tests on hourglass specimens have been discarded and only tests on parallel-sided specimens with axial extensometers have been used in the assessment. Some laboratories have used diametric extensometers for the initial test setup or have doubled their tests with axial extensometers, and these tests have been included.

Presently the database includes:

- 600 product records
- 486 composition records
- 1612 tensile test records
- 1998 impact test records, including 184 impact plots
- 351 fracture toughness test records
- 350 creep test records
- 439 fatigue test records, including tests with hold times

All above databases are linked to each other and their records include testing procedures and thermo-mechanical treatments applied from ingot to final specimens, as well as ageing and irradiation data. Irradiation doses entered in the database go up to 70 dpa and thermal ageing times to 30000 h. A summary database summarizes all above properties in a single page view and sorts them out by ingot, by product or by sub-product.

Results of the tests performed on specific products, e.g products with different boron concentrations to test the effect of helium [24], are used in the discussion but not in the design allowable determination.

4. RCC-MRx

RCC-MRx will cover, in addition to fission reactors materials, fusion reactor materials. This includes already type 316L(N)-IG for ITER vessel and in-vessel components. A new materials properties group has been tentatively given to Eurofer (A3.19AS) in Section III, Tome 3, sub-section Z. This designation is used in Eurofer RCC-MRx files.

The following Eurofer files are currently examined by the RCC-MRx expert groups.

- DMRx-A3.GEN modification requests to Tables A3-GEN.22 and A3-GEN.1b
- DMRx 242-6-Forged bars, DMRx 243-3-Plates and DMRx 244-3-Tubes.
- RM Procurement specifications RM 242-6, RM 243-3 and 244-3 for Eurofer steel.
- DMRx-A3.19AS Materials properties data.
- A3.19A Materials Procurement support document for Eurofer steel.
- A3.19AS Materials properties data support document for Eurofer steel (Appendix A).

The first two files are administrative modification requests that specify types of the Eurofer products. The RM procurement file gives detailed specifications for each type of product. The Materials properties file gives design allowable values as they should appear in the codebook. The last two files are support files. These support files are not public and provide detailed justifications for each recommendation made in the materials properties data file. The support files include experimental data used to determine the allowable values. The actual databases, however, are accessible only to the laboratories that have contributed to the data bank. Nominative authorization may be given to individuals after case-by-case examination.

5. Materials properties

The full analysis of materials properties data is beyond the scope of this paper. Only those related to fatigue, thermal creep and creep-fatigue are discussed below.

5.1. Fatigue properties

Fatigue properties are covered in RCC-MRx under analysis data [1]: cyclic curves, values of K_ε, K_v and Ks, fatigue curves, fatigue crack growth rate.

The information needed to treat all these items go beyond conventional fatigue data reported for most labs, e.g. total strain range versus number of cycles to failure (Fig. 1). Preferably full recording of the evolution of hysteresis loops and evolutions of the maximum and the minimum stresses during fatigue life are needed. These values are particularly important for steels like Eurofer that show cyclic softening even in the irradiated state (Fig. 2).

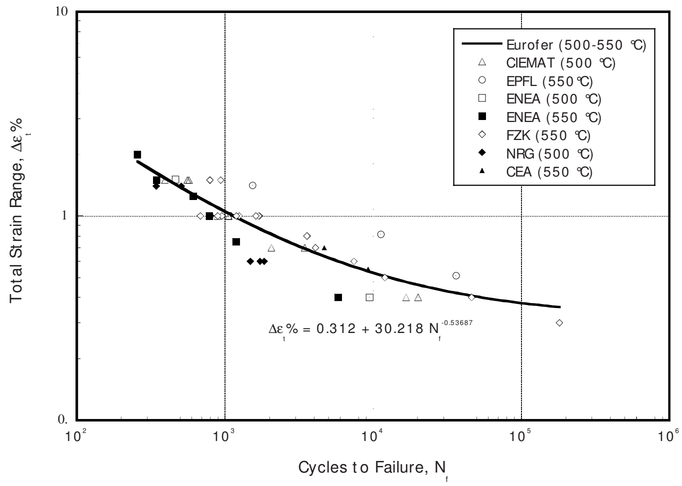

Fig. 1. Comparison of irradiated and unirradiated Eurofer fatigue results.

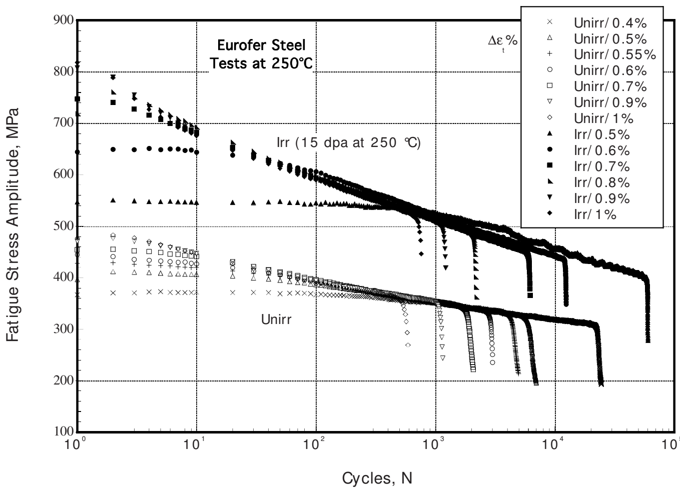

Fig. 2. Cyclic softening of Eurofer steel in unirradiated and irradiated states (data curtsey of E. Materna-Morris of KIT/Germany from SPICE experiment).

For some items like symmetrization coefficient Ks, specific tests are needed. Actually, the three types of tests[‡] usually performed for determination of Ks can be extracted from the available Eurofer data but they lack precision or are performed at high strain ranges irrelevant to design. Figure 3 shows the tentative recommendation made for Eurofer Ks (Ks=0.5).

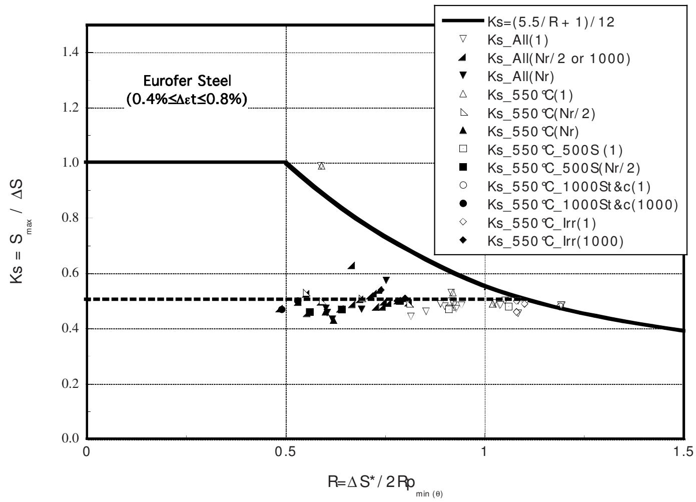

Fig. 3. Tentative Ks recommendation for Eurofer (solid line from RCC-MR for 316L(N) steel).

5.2 Creep properties

The first appearance of creep in RCC-MRx materials properties data file is under borderline section. Here, negligible creep domain is defined as $\varepsilon_{creep} < 0.05\%$ under a stress equal to 1.5 S_m. For Eurofer steel, at temperatures ≥ 500°C times to reach 0.05% strain is very short (<40 h) and practically one has to consider thermal creep for all components operating at such temperatures. Only at 450°C and below negligible creep times reach 10^5 h and over. Long time creep tests at low temperatures (e.g. 400°C) are needed for more accurate determination of the lower temperature bound.

The second appearance of creep is under analysis data 2: primary creep, secondary creep, tertiary creep, creep strain rules, creep rupture, values of S_t, fatigue-creep interaction diagram, maximum allowable strain and D_{max}. Most laboratories have reported only the basic creep information, e.g. stress rupture, time to 1% creep deformation, time to tertiary creep and secondary creep rate. For full RCC-MRx analysis, this is inadequate. Digital recording of creep curves and in particular determination of the end of primary creep (time and strain) are needed.

Figure 4 shows the master curve established for creep rupture of Eurofer. Such master curves have been established also for time to 1% creep deformation and the onset of tertiary stage, as well as the creep rate. From these master curves average and minimum curves have been derived and used for determination of stress intensity criteria such as Sr and St.

[‡] The 3 types of test are :
- Type A tests: Cycling at the strain range $\Delta\varepsilon_t$ after monotonic pre-straining to $\varepsilon^{initial}$.
- Type B tests: Cycling at $\Delta\varepsilon_t$ after initial pre-cycling at $\Delta\varepsilon_t^{initial}$.
- Type C tests: Cycling between 0 and $\Delta\varepsilon_t$ strain.

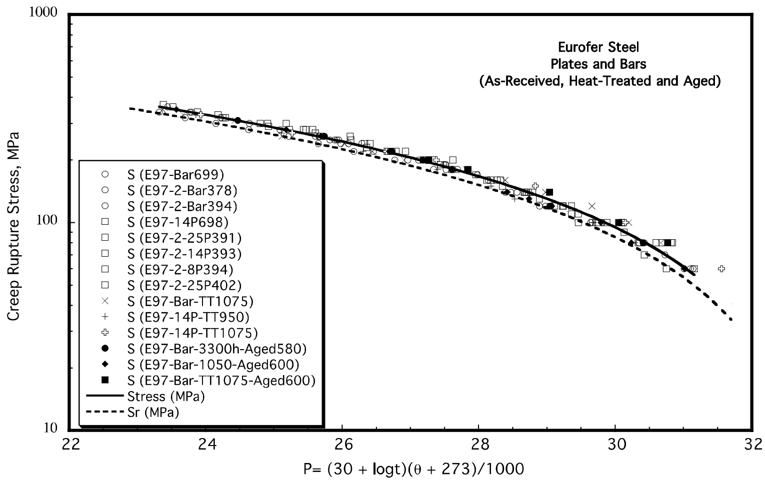

Fig. 4. Average and minimum master creep rupture curves for Eurofer, including results after additional solution heat treatments and aging.

Limited information available on actual creep curves have been used for determination of primary creep law and plots of isochronous curves.

5.3. Creep-fatigue

Creep-fatigue interaction analysis is already difficult for materials such as austenitic stainless steels that show saturation in hardening after the first few cycles. It is even more complicated for martensitic steels that show cyclic softening, and different peak stresses, throughout the fatigue life (Fig. 2).

For ITER and TBMs, creep-fatigue interaction is best studied through fatigue tests with hold times, representing the pulsed operation mode. Presently, Eurofer database contains strain controlled and thermal fatigue tests performed on unirradiated materials with hold times up to 1 h, in tension or in compression or in both sides of the cycle. It also contains continuous fatigue tests results on specimens irradiated up to 15 dpa, and creep-fatigue test results on specimens irradiated up to 2.5 dpa.

Unfortunately, many of the laboratories performing these tests have not fully reported the evolution of peak and minimum stresses with number of cycles or stress relaxation curves during hold times. To support Eurofer evaluation, results from in-house tests [25] on Mod 9Cr-1Mo specimens have been integrated in the assessment. The latter tests are performed at ±0.3% strain amplitudes with hold times of 30 min. and 90 min. in tension or in compression.

Each stress relaxation cycle can be fully represented by eq. (1):

$$Sr = (Smax - So)\ Exp\ (-ATmP) + So \qquad (1)$$

Where S_r is relaxation stress (MPa), S_{max} is the peak stress, in tension (S_{tmax}) and in compression (S_{cmax}), A is a parameter dependent on fatigue strain range, T_m is hold time, P exponent and So constant. For 30 minutes hold in tension the equation at half life is S_r = (233.28-134) Exp(0.26042*$T_m^{0.2696}$)+134 with T_m in seconds. Relaxation cycles at a given strain range for hold times of 30 min. and 90 min. do not differ much. Their forms are slightly different in tension and in compression.

Ideally, for creep damage evaluation, each relaxation curve should be divided into small time fractions, average stress for each time fraction calculated and used to find the average times to rupture for each time fractions, and finally creep damage for each time fraction calculated from Δt_i/tr. Without such steps, using the peak stress at the beginning of each cycle overestimates the creep damage and using the stress at the end of cycle underestimates creep damage.

Adding creep damages calculated for each time fraction gives creep damage for that cycle, summing up these from cycle N=1 to cycle N=Nr gives the total creep damage for each test.

However, this method is time consuming and often unrealistic for all fatigue cycles. A simpler approach can be used based on the following observations.

(1) Cyclic softening is less pronounced at lower strain ranges that are more pertinent to design (see e.g. Fig. 2). The ratio of the minimum stress (at the end of relaxation) over the initial stress (at the beginning of relaxation) remains practically constant throughout the fatigue life, even at high strain ranges. As a result, if the relaxation cycles are plotted using normalized stress (S_r/S_{max}) versus time, they almost converge and a mean representative cycle curve can be derived for all cycles of a given experiment, see e.g. Fig. 5.

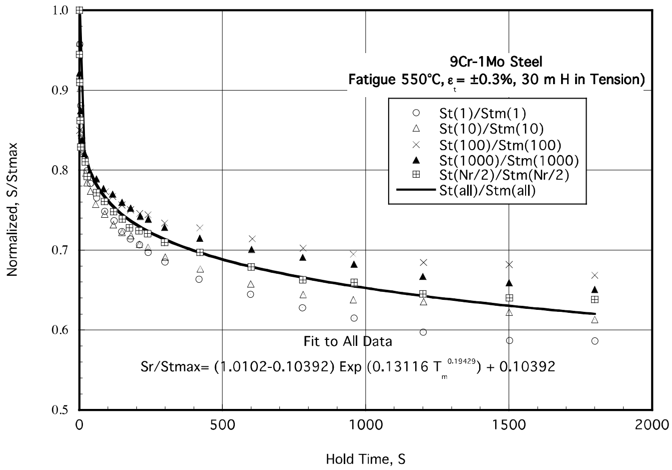

Fig. 5. Determination of mean relaxation cycle using normalised Sr/Stmax.

(2) When creep damage calculated for individual cycles is plotted versus number of cycles, not only one notices that most of the damage occurs during early cycles but also that the evolution of creep damage v. N on log-log plot is linear. In the case of 9Cr-1Mo steel test with 30 m hold, we have calculated the total creep damage using all cycles: Dc= 0.0645. The linear softening evolution on log-log can be used to find an equivalent cycle that when multiplied by the number of cycles gives the same Dc.

(3) Using the exact relaxation cycle form at cycles 1, 10, 100, 1000, Nr/2 and near Nr (4478), just before fast drop in stress, we have calculated the damage at these cycles (Table 2). If these are multiplied by the number of cycles to near rupture, one notices that cycle 1 over-estimates the damage and cycle Nr/2 under estimates the damage. A cycle between cycles 100 and 1000 should represent the average damage cycle that when multiplied by 4478 gives damage close to 0.0645. By further exploring these observations, we have determined this average cycle to be around log (N) = 0.7 log (Nr).

Table 2. Total creep damage (0.0645) for this test [Mod 9Cr-1Mo, T=550°C, ε_t= ±0.3%, HT 30 m, Nr=4802], can be obtained by Dc (cycle) at 100<N<1000, times Nr (near rupture).

Cycle Number	D_c (cycle)	Nr*D_c (cycle)
1	0.004476233	20.044 (excessive)
10	0.000584296	2.616 (excessive)
100	0.00012879	0.576 (just over)
1000	1.36E-05	0.0608 (just under)
2400 (Nr/2)	3.93E-06	0.0176 (far below)
4478 (before drop)	8.02E-07	0.0036 (far below)

We have then used this average representative stress relaxation cycle form to calculate D_c. D_f is obtained from the ratio of Nr (with hold) /Nr (without hold), Fig. 6.

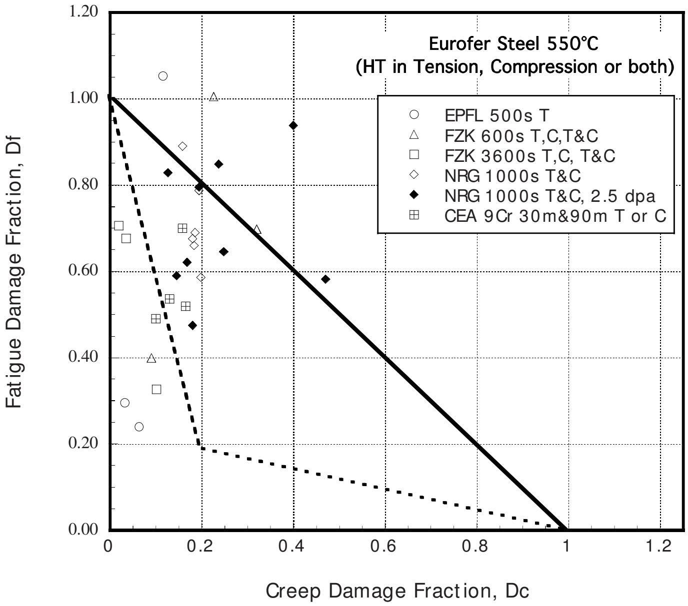

Fig. 6. Tentative creep-fatigue interaction diagram recommended for Eurofer.

Most cumulative creep-fatigue damage data in Fig. 6 fall close to the fatigue damage axis. This is due to the fact that hold-time durations used here are short, and creep-fatigue damage is dominated by fatigue. Some data not only fall close to the fatigue axis but below $D_f = 1$. This is partly due to the fact that in our treatment preference is given to the laboratory reference test results for Nr (without hold) and most Labs. have performed only one or 2 reference tests. If the reference test result falls in the upper part of the scatter band, then fatigue damage will be overestimated. Another source of scatter is the test environment, particularly for tests with hold times in compression. When these tests are done in air, compression hold tests, in general, give higher damage. Limited tests done in vacuum show there is no difference between tension and compression hold results [26]. The creep-fatigue damage based on a central point at (0.2 ; 0.2) adequately covers most experimental points in Fig. 6 and is recommended for Eurofer. It should be added that if the actual fatigue design curve is used for damage calculation (incorporating coefficients of 2 on total strain range and 20 on number of fatigue cycles to failure), all experimental points will fall well above the D=1.

6. Conclusions

Eurofer code qualification files have been submitted to RCC-MRx. At first, these files are proposed for plates, tubes and forgings to be used in EU Test Blanket Modules. They will be expanded in the near future for full code qualification. The ultimate goal is to qualify the steel for application in DEMO and fusion power reactors.

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