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Current status and recent research achievements in ferritic/martensitic steels

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ABSTRACT

When the austenitic stainless steel 316L(N) was selected for ITER, it was well known that it would not be suitable for DEMO and fusion reactors due to its irradiation swelling at high doses. A parallel programme to ITER collaboration already had been put in place, under an IEA fusion materials implementing agreement for the development of a low activation ferritic/martensitic steel, known for their excellent high dose irradiation swelling resistance. After extensive screening tests on different compositions of Fe–Cr alloys, the chromium range was narrowed to 7–9% and the first RAFM was industrially produced in Japan (F82H: Fe–8%Cr–2%W–TaV). All IEA partners tested this steel and contributed to its maturity. In parallel several other RAFM steels were produced in other countries. From those experiences and also for improving neutron efficiency and corrosion resistance, European Union opted for a higher chromium lower tungsten grade, Fe–9%Cr–1%W–TaV steel (Eurofer), and in 1997 ordered the first industrial heats. Other industrial heats have been produced since and characterised in different states, including irradiated up to 80 dpa. China, India, Russia, Korea and US have also produced their grades of RAFM steels, contributing to overall maturity of these steels. This paper reviews the work done on RAFM steels by the fusion materials community over the past 30 years, in particular on the Eurofer steel and its design code qualification for RCC-MRx.

1. Introduction

Fusion structural materials activities have evolved over the past four decades, maturing with time and becoming design oriented and lately achieving code qualified status [1]. At the beginning, in the early 80s, the activities were performed at laboratory or national levels and often on small melts. With the start of international collaborations, such as for the NEXT machine in Europe, these activities began to converge and follow a common objective. With the ITER agreement, fusion materials activities were separated in two branches, one following ITER objectives and other dealing with beyond ITER (DEMO and Power Reactors). The latter activities were mostly grouped under an IEA fusion materials implementing agreement/Annex II; less formal and more open to new partners than ITER, although the initial members were the same as ITER.

Early inputs to ITER and IEA activities were largely brought from the fission program [2,3]. In fact, most of the fusion material specialists were former fission specialists that had collaborated under different fission international programs, e.g. the European Fast Reactor (EFR) program. However, fusion established from the beginning its distinctive mark «Low Activation» and with the lull in fission activities became the driving force for development of structural materials for nuclear applications.

ITER materials activities were largely influenced by its time schedule. Advanced low activation materials such as vanadium alloys and SiC_f/SiC composites, and even martensitic steels were discarded in favour of the more robust stainless steel type 316L(N), with proven service experience in several generations of Fast Breeder Reactors (FBRs) [2]. In fact, 316L(N)-IG chosen for ITER is a derivative of 316L(N)-SPH used in the Superphénix and retained for the EFR [2,4]. DEMO materials activities did not have the same time constraint and in addition it was well known that the solution annealed austenitic stainless steels such as 316L(N) are not suitable for high dose applications (in DEMO > 70 dpa) due to their irradiation swelling [2,5]. Vanadium alloys and SiC_f/SiC composites developments lasted several years under the IEA

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collaboration but finally were marginalised due to several unresolved shortfalls (e.g. low temperature irradiation embrittlement and absence of reliable protective coating for vanadium alloys [6], and low thermal conductivity and low fracture toughness for SiC_f/SiC [7]). The bulk of IEA collaboration from the beginning went on the martensitic steels [8] with precursor work in several member countries, e.g. Manet and Optifer in EU [9,10].

This paper presents the work done from early stages of RAFM steels development to their more recent qualification for reactor design codes [11–20]. Aspects related to higher helium to dpa generated in fusion environment are left out since they are still under investigation and not ready to be integrated in design codes. An excellent review of the effects of helium in irradiated structural alloys is given in [21].

2. Materials

As mentioned in the introduction, initial inputs to fusion program were strongly influenced by the fission experience. Nevertheless, extensive screening tests were performed on different Fe–Cr compositions before narrowing down the chromium range to 7–9% and finally converging towards a composition similar to that of the conventional Modified 9Cr–1Mo steel. Fig. 1 shows an example of the results after irradiation in FFTF at 365 °C to 7 dpa, where the changes in DBTT of 7–9%Cr alloys are smaller (results compiled from presentations at an IEA topical meeting).

In 1995, IEA/Annex II defined a reference low activation steel to be produced in Japan and characterised by all members. The term RAF/M (Reduced Activation Ferritic/Martensitic) steel was used instead of LAF/M (Low Activation F/M Materials) to distinguish these grades from the ultimate low activation materials that will push concentrations of high activation residual elements even lower. Two industrial heats were fabricated (Fig. 2) and designated IEA-F82H heats, to distinguish them from an earlier F82H heat, also produced in Japan (called Pre-IEA F82H) [10].

The IEA heats were characterised by all partners and the results were collected and after validation entered in dedicated relational databases that were then used to derive F82H design allowables. In parallel, several IEA partners continued work on other RAFM grades including JLF1 and JLF2 in Japan [13]. In 1997, EU opted for a higher chromium and lower tungsten composition grade, Eurofer (also called Eurofer97), to improve corrosion resistance and neutron efficiency [14]. From the beginning an industrial

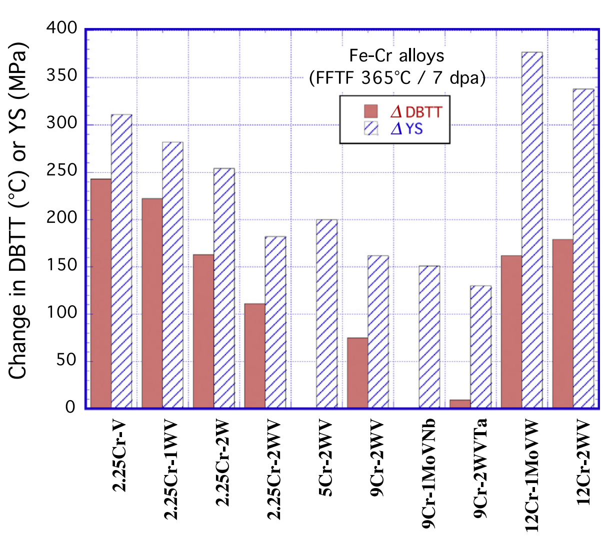


Fig. 1. Effect of irradiation on ductile-to-brittle transition temperature (sub-size specimens) and yield stress of Fe–Cr alloys.

specification was used for production of Eurofer steel [22]. Other countries, China (CLAM: Chinese Low Activation Material), India (INRAFM: Indian Reduced Activation Material), Korea (ARAA: Advanced Reduced Activation Alloy) are investigating similar compositions and targeting industrial productions for ITER TBMs. Limited work has also been going on in Russia (RUSFER: Russian Reduced Activation Material). United States and in particular ORNL have been pursuing basic studies to develop RAFM steels with higher strength and improved radiation resistance with applicable temperatures up to 600–650 °C. Additional information of above work can be found in [14–20].

Table 1 presents chemical compositions of four RAFM steels produced in different countries along with the composition of the conventional Modified 9Cr–1Mo steel. The basic difference between RAFM steels and the conventional steel is in replacement of Mo and Nb with their equivalent low activation elements (W and V) [14]. Other high activation residual elements are kept as low as possible. Tantalum is added for grain size control.

Heat treatment specifications and acceptance values of RAFM steels are similar to the conventional 9Cr–1Mo steels [3]. However, while Modified 9Cr–1Mo steel has long been design code qualified, most of the RAFM steels are still in the development stage, except F82H and Eurofer steels. Eurofer achieved code qualification status after 30 years in 2013 with its entry in RCC-MRx edition 2012 (under Section III, Tombe 1, Sub-Section Z and denomination A3.19AS)[1]. F82H is expected to follow with its entry in the Japanese codebook.

3. Databases

An important distinction is to be made between general material's properties data and the code-qualified materials properties data [23–27]. In the case of code-qualified properties, all data collected must be harmonised and validated by expert groups before they are entered in the databases. In addition, each datum point must be obtained according to an internationally accepted procedure and should be fully traceable back to its origin and experimental and testing history. For instance, all specimens taken from F82H sub-products dispatched to laboratories are linked to the production route shown in Fig. 2.

In the early stages of design, e.g. conceptual design analysis, the above requirements were relaxed and missing information about some properties were taken from not fully qualified sources. However, in the detailed design analysis stage and later for engineering design analysis, all data used were "code-qualified". As a result, the quality of materials properties data collected in the fusion program has varied with time. For instance, materials properties data from the literature were initially included in the ITER MPH (Materials Properties Handbook) to allow conceptual design analysis to proceed [4]. For the ITER Interim Structural Design Analyses (ISDC) only the code-qualified part of the data was used [28]. Recent updates of MPH have been harmonised with SDC-IC (formerly ISDC) and contain mostly code-qualified data. For a fuller insight into the procedures required for code qualification, the reader is referred to EU CEC works carried out on different properties of 316 and Mod. 9Cr–1Mo steels under EFR contracts and used to revise RCC-MR design allowables. An example of this is available in [29] for fatigue properties.

All Eurofer data entered in the EU databases are now code qualified. Extracts from these databases are used at first to derive design allowables for ITER Test Blanket Modules (TBMs), since TBM design is in an advanced stage, its construction schedule is close (2020) and does not require irradiation data higher than 3 dpa. For DEMO

[1] RCC-MRx is a new French reactor construction code combining RCC-MR and RCC-Mx and includes fusion materials.

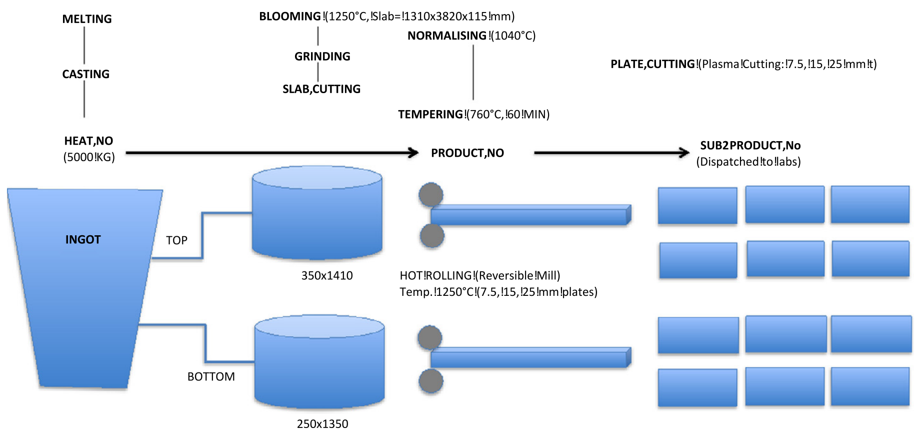


Fig. 2. F82H production route and distribution.

Table 1
Nominal chemical compositions of several RAFM steels and conventional Mod. 9Cr–1Mo steel.

	Mod. 9Cr–1Mo	F82H	Eurofer	INRAFM	CLAM
Cr	8.5	7.7	9	9	9
C	1	0.09	0.11	0.11	0.1
Mn	0.4	0.16	0.4	0.5	0.45
P	<0.020	0.002	<0.005	<0.002	
S	<0.01	0.002	<0.005	<0.002	
V	0.21	0.16	0.2	0.22	0.2
B		0.0002	<0.001	<0.001	
N2	0.05	0.006	0.03	0.03	
O2		<0.01	<0.01	<0.01	
W		1.94	1.1	1.35	1.45
Ta		0.02	0.07	0.07	0.15
Ti		100 ppm	<0.01	<0.005	
Nb	0.08	1 ppm	<0.001	<0.001	
Mo	0.92	30 ppm	<0.005	<0.002	
Ni	<0.20	200 ppm	<0.0050	<0.005	
Cu	<0.10	100 ppm	<0.0050	<0.002	
Al	<0.040	30 ppm	<0.0010	0.005	
Si		1100 ppm	<0.050	<0.05	
Co		50 ppm	<0.0050	<0.005	
Sn		(<20 ppm)			
As		(<50 ppm)	As + Sn + Sb + Zr < 100 ppm	AS + Sn + Sb < 0.03	

and power reactors, where doses are much higher, design is still evolving and effects of irradiation damage are more complicated. Furthermore, some experimental data generated and analysed are still for understanding the irradiation damage, particularly the effects of higher helium formation under 14 MeV neutrons and its synergistic effects when combined with dpa.

More recent articles on Eurofer steel development are given in [30–46]. References in these articles can be used to locate other articles.

Presently the EU RAFM database has (excluding data from on going work):

- 672 Product records out of which 218 are for Eurofer steel.
- 602 Composition records out of which 151 are for Eurofer steel.
- 1535 Tensile records out of which 762 are for Eurofer steel.
- 2154 Impact records out of which 1071 are for Eurofer steel.
- 202 Impact plots records out of which 82 are for Eurofer steel.
- 505 Fracture toughness records out of which 445 are for Eurofer steel.
- 350 Creep records out of which 227 are for Eurofer steel.
- 438 Fatigue and creep-fatigue records out of which 328 are for Eurofer steel.

A multi-platform proprietary Runtime solution is written for the above databases that runs on different operating systems as well as on the hand held devices. Each database in this solution has at least 3 views: Search, List and Detail. An example of the detail view for fatigue test results is shown in Fig. 3. The buttons on the right hand side in this view have their usual meanings and those above are links to other databases. Two of the databases, Impact and Fracture, have sub-databases for depicting impact plots and master curves. A Summary database, button shown at the top right corner in Fig. 3, allows visualisation of all results obtained on a given heat, or product or sub-product on a single page. The Runtime solution also incorporates links to design and support documents derived from the database results.

4. Code qualification

In the period 2011–2013, detailed design documents were prepared for Eurofer steel and submitted to AFCEN for Eurofer entry as a new structural material in the RCC-MRx. These have been partially approved since and Eurofer has entered in an approbatory status in Tome 6, RPP3-2012 of RCC-MRx. The purpose of approbatory status is to allow industry and interested parties to comment the files before they are made permanent [1].

The first files written for Eurofer code qualification were materials procurement files. These files had to be: (a) sufficiently detailed to allow any manufacturer to bid for fabrication and (b)

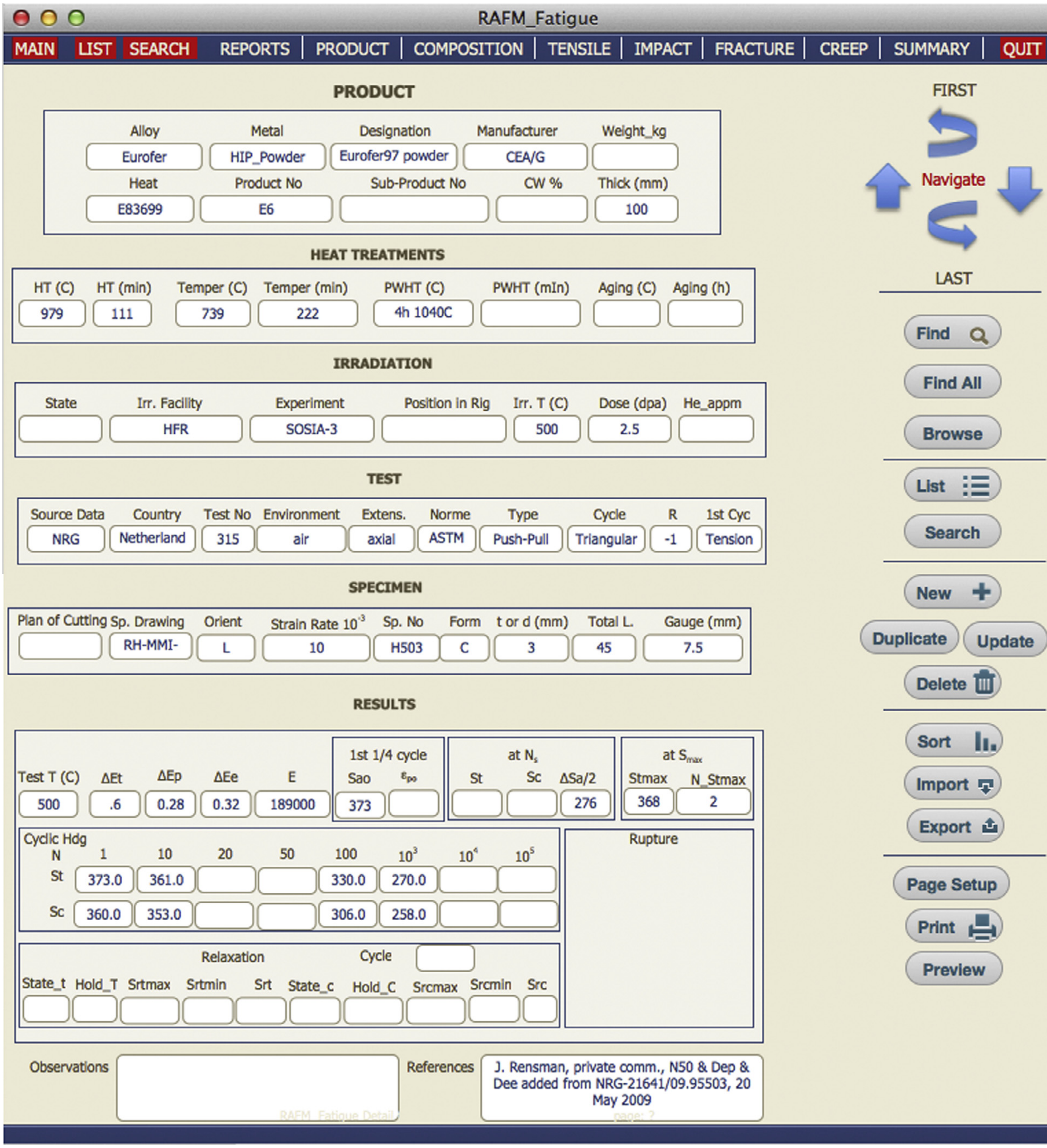


Fig. 3. Screen dump of RAFM fatigue detail view database.

adequately precise to ensure that the end products, irrespective of their producer, would comply with the specified properties. In fact, during the code qualification, a minimum number of industrial heats (3–5) must be characterised to cover the effect of heat-to-heat variations.

Materials procurement files submitted for Eurofer steel covered plates, tubes, bars and forgings. Welding specifications are yet to be finalised [45,46]. All the base metal files were backed up with support documents that include results of inspections, examinations, macro and microstructural examinations, hardness, etc. for each product. However, only the plate products were approved for RCC-MRx 2012 edition.

For plates, tubes, bars, forgings produced from an ingot, information relevant to manufacturing procedures, thermo-mechanical treatments, etc. were registered. This also included inspections, examinations, compositions and control tests performed by manufacturer and laboratories. Sub-products cut from large products received from the manufacturer and dispatched to different laboratories[2] were referenced, see e.g. Fig. 2. Each specimen cut from a sub-product was also fully identified with regard to its position and orientation inside the sub-product. As a result, each specimen in the database can be fully traced back to its thermo-mechanical history and source ingot.

Notice that special attention has been paid to machining of some specimens. This is in addition to the usual specifications

[2] These laboratories in general have participated in round robin test programs and are qualified for testing.

dealing with specimen geometry and testing procedures (e.g. ASTM standards). For instance, in the case of fatigue only proven sub-contractors were used and specimen surface finish was rigorously controlled. Only results from laboratories qualified during round tests have been accepted. Furthermore, only parallel-sided specimens with axial extensometry were accepted. Diametric extensometry results were accepted if they were duplicated by a second test without extensometry or if the extensometry blades used were qualified for fatigue testing. If a specimen was aged or irradiated prior to testing, all relevant information were entered in the database along with the specimen number, testing conditions, laboratory test number and its references, Fig. 3. A link parameter was derived from: heat number, product number and sub-product number, to link test results databases to products and compositions databases.

The second set of files written for RCC-MRx was materials properties data files. These are organised, as in the RCC-MRx, in 5 sections:

– Physical properties.
– Borderlines.
– Analysis data 1.
– Analysis data 2.
– Analysis data 3.

Physical properties required for conventional design have been evaluated for F82H and Eurofer steels [10,30]. They include: coefficients of thermal expansion (average and instantaneous), modulus of elasticity, Poisson's ratio, density, specific heat, thermal conductivity and thermal diffusivity. For the special case of fusion, magnetic and electrical properties have also been evaluated.

Physical properties of RAFM steels are similar to those of the conventional 9Cr–1Mo steels. In fact, in the beginning when a property was missing for RAFM steels that of the 9Cr–1Mo was used. Even today, for some physical properties of Eurofer, not yet fully evaluated, results of 9Cr–1Mo and F82H are tentatively used.

Borderlines define conditions where certain analyses are not needed. For instance, thermal creep analysis is not required if the creep deformation does not exceed 0.05% (negligible creep) under a stress equal to 1.5 S_m at the service temperature. Precise evaluation of negligible creep requires long duration creep tests at low stresses. RAFM steel data are still insufficient for its precise evaluation. In the meantime, based on the available results, one can reasonably assume that thermal creep can be ignored at temperatures below 400 °C. At temperatures higher than 500 °C, time to reach 0.05% creep is very short and thermal creep cannot be ignored even in the ITER like pulsed operation mode.

Likewise, negligible irradiation creep limit is defined as a dose below which irradiation creep deformation does not exceed 0.05% under a stress equal to 1.5 S_m. For ITER TBMs, where the maximum dose is less than 3 dpa, irradiation creep can be effectively ignored except for bolts. Irradiation creep in general does not provoke physical damage and can even be beneficial e.g. residual stress relaxation in welded joints. However, in specific cases, such as bolts, even very small deformations can cause significant reduction is the tightening stress.

At higher doses irradiation creep cannot be ignored [47] and its effects should be added to those of the thermal creep and irradiation swelling. Irradiation swelling does provoke physical damage and at levels higher than 3% in volume (1% in dimension) needs special design procedures. At above 6% it is beyond design and not tolerated. Fortunately, martensitic steels show excellent resistance to swelling and even at DEMO dose levels their swelling is expected to remain low. Possible effects of higher helium on swelling resistance of martensitic steels are yet to be fully determined.

Analysis data 1 deals with time independent properties such as tensile, impact, fatigue and fracture. Tensile strength data were used to derive design stress intensity values S_m and S. Tensile ductility and toughness values were used to ensure that Eurofer has adequate resistance to brittle fracture. Fatigue results, mostly from continuous low cycle fatigue testing but also some high cycle and thermal fatigue results, were used to derive fatigue design curves, applying default knock down factors of 2 on strain and 20 on number of cycles to failure.

Tensile properties of RAFM steels [48] are close to those of the conventional Mod. 9Cr–1Mo steel and are higher than those of the SS 316L(N) steel at temperatures less than 500 °C, Fig. 4. At higher temperatures, both RAFM and conventional martensitic steels, lose their strength and are seldom recommended for service at above 550 °C. Other grades of these steels, such as oxide dispersion strengthened steels (ODS), are developed to increase their temperature window [21].

Impact and fracture toughness values of RAFM steels in the as-received condition are quite high and not of concern in design. These steels show a Ductile-to-Brittle Transition Temperature (DBTT) at $\leqslant -50$ °C. As shown in Fig. 5, Eurofer DBTT is slightly lower than those of the 9Cr–1Mo and F82H. The fracture toughness of RAFM steels in the ductile region is very high, e.g. J_Q of Eurofer at 60 °C is in the range of 300–620 kJ/mm^2.

Fatigue is the only time independent property of major concern in time independent properties because of continuous cyclic softening and breakdown of martensitic microstructure, Fig. 6 [49]. In fact, due to cyclic softening tensile resistance of martensitic steels can become lower than the 316L(N) steels even at low temperatures, rendering their time dependent damage calculation more difficult.

Analysis data 2 deals with time dependent properties such as creep and creep-fatigue [50,51].

Creep results of Eurofer steel were used to determine stress to rupture, stress for time to reach 1% deformation and stress to reach the onset of tertiary creep criteria. Combination of these criteria was used to determine the stress intensity criterion S_t. On a finer scale; creep deformation curves were used to plot isochronous curves. Creep-fatigue interaction data were generated mostly from fatigue with hold times in tension, in compressing or both sides of the cycle. These results were used to plot creep-fatigue interaction diagrams. Limited sequential creep-fatigue and fatigue-creep tests were also used to determine K_s.

In comparison with stainless steel, conventional and RAFM martensitic steels have a lower creep resistance at 550 °C and higher. Amongst the martensitic steels, Mod. 9Cr–1Mo steel has higher creep strength than RAFM steels, probably due to its higher

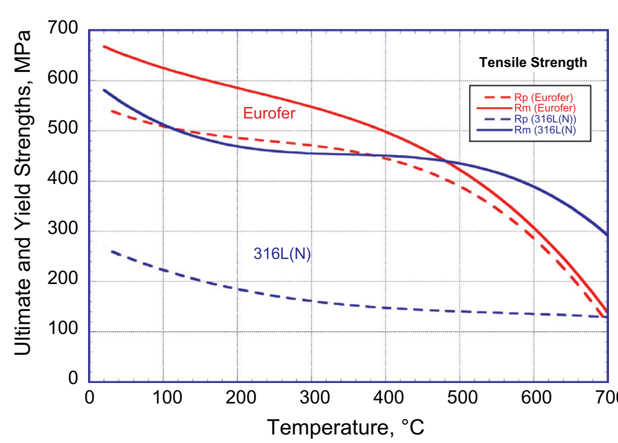


Fig. 4. Comparison of tensile strengths of 316L(N) and Eurofer.

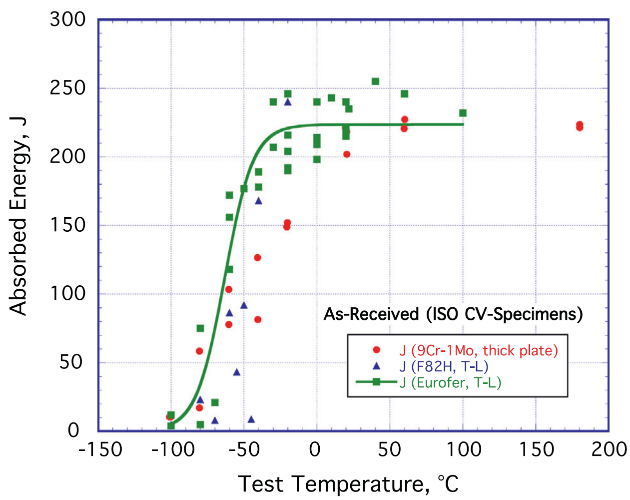


Fig. 5. Charpy impact test results for Mod. 9Cr–1Mo and RAFM steels.

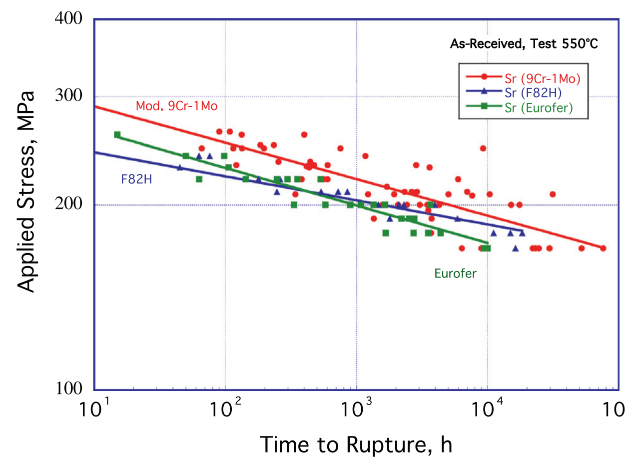


Fig. 7. Creep rupture properties of Mod. 9Cr–1Mo and RAFM steels at 550 °C (all in the as-received state).

residual elements and larger average (austenitic) grain size, Fig. 7. Present RAFM creep test durations may not be sufficiently long (although some F82H tests have reached 120,000 h) for reliable extrapolation to nuclear reactor design time of > 300,000 h, but are adequate for TBM and early DEMO operations. Trend curves obtained from extrapolation of RAFM creep rupture data are anyhow compatible with those obtained for the Mod. 9Cr–1Mo with rupture times greater than 230,000 h [49].

Cyclic softening concern is aggravated during creep-fatigue with hold time (Fig. 8) and there are additional concerns about a possible reduction in creep strength of cyclic softened materials [49]. This makes creep-fatigue damage calculation even more complicated [50]. Depending on what stress is used for creep damage calculation during fatigue life, e.g. at first cycle, at half-life, etc. one can overestimate or underestimate the creep damage.

Analysis data 3 deals with irradiation effects on time independent and time dependent properties. Modified 9Cr–1Mo steel is used essentially in conventional power plants or steam generators of nuclear power plants, i.e. in unirradiated zones. When it is used in irradiated zones, it is in fuel sub-assemblies that operate at high temperatures and are replaceable thin section components. As a result, analysis data 3 mostly relies on RAFM steel irradiation data, aggravated by higher helium to dpa generations.

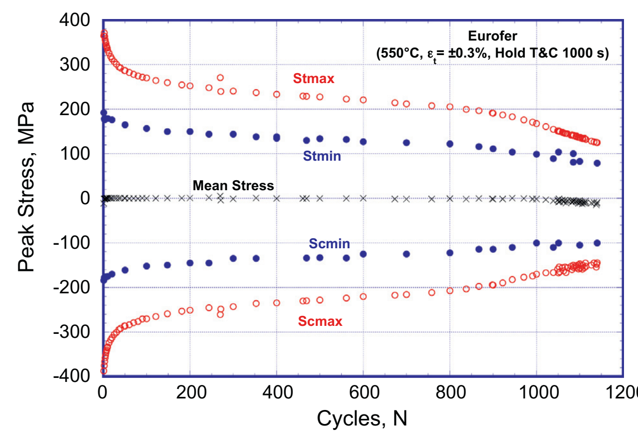
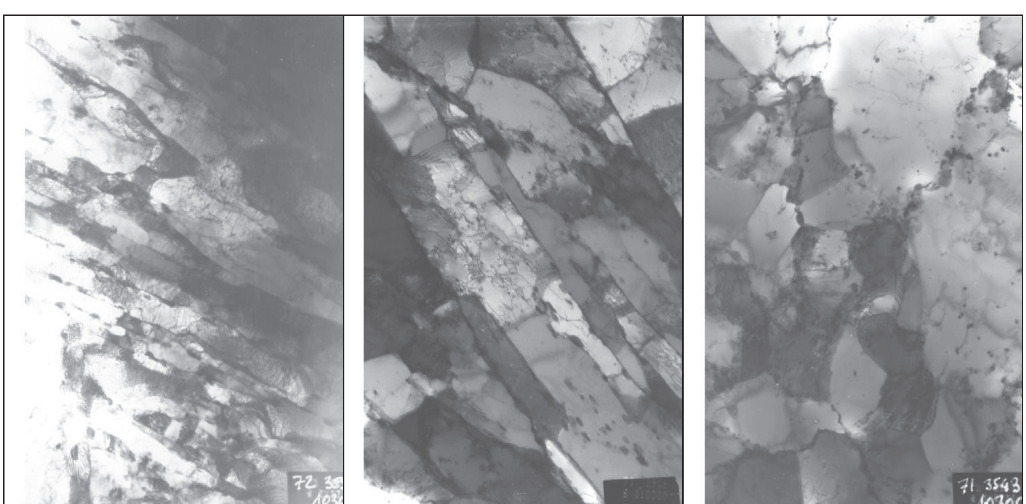


Fig. 8. Evolution of stress amplitude during fatigue cycling with 1000 s hold in tension and in compression (Eurofer, unirr and irr, 250 °C).



Initial state	Pure fatigue (ε_t=±0.3%, 550°C)	Fatigue-relaxation (+t_h=90min)

Fig. 6. Microstructures of conventional 9Cr–1Mo steel in initial, fatigued and fatigue with 90 min hold states.

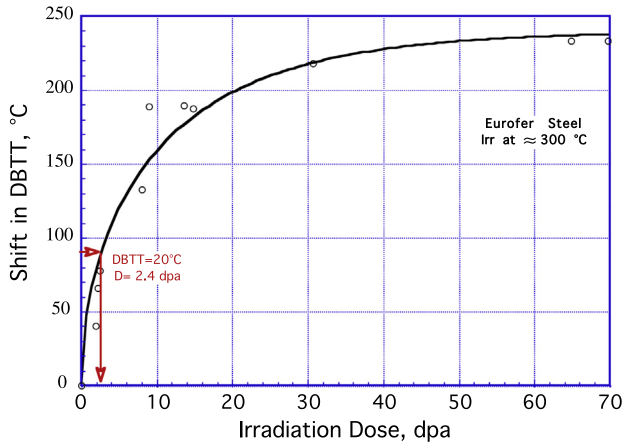


Fig. 9. Change in DBTT of Eurofer steel versus irradiation dose at 300 °C.

The results obtained for RAFM steels show that despite progress in improvement of low temperature irradiation embrittlement, these steels cannot be recommended at present for service at doses higher than 3 dpa at $T < 350$ °C, Fig. 9. The same results show that at irradiation temperatures higher than 350 °C the toughness of RAFM steels remains adequate even at much higher doses, unless future irradiation experiments under fusion relevant neutron spectra prove otherwise.

Cyclic softening observed in unirradiated state also occurs in the irradiated state [42]. Under these conditions, calculation of creep-fatigue interaction diagram [50,51], already difficult in the unirradiated state, becomes even more difficult. Furthermore, since most of the results are from fatigue tests with relatively short hold times (<1 h relevant to TBM and early DEMO); they do not cover the higher creep damage region relevant to DEMO operation.

5. Discussion

Qualification of the Eurofer steel for RCC-MRx is an important achievement for the fusion materials community. However, full qualification for a specific application goes beyond simple materials properties analysis. For instance, depending on the manufacturing technique used, specific types of joints may need to be qualified. In the case of ITER and ITER TBMs, conventional welding processes (TIG, MIG, Laser, plasma, EB, Hybrid), as well as solid and powder HIP processes have been considered [52]. Development of these joints has to be accompanied with non-destructive techniques for prior and in-service inspections. Furthermore, depending on the type of cooling media employed, the materials properties may need to be evaluated in those media. In fact, the cooling media may limit the service parameters rather than the mechanical properties. For instance, if the liquid metal is used for cooling, the upper temperature of the Eurofer steel may be fixed by the corrosion/erosion rate of the steel [53].

The above considerations demonstrate the time and resources needed for the development and qualification of a new structural material in nuclear industry. The expertise and facilities involved often require extensive international collaborations. The ITER and IEA collaborations are two good examples of this. Production of F82H steel in Japan under an IEA implementing agreement and sharing it with partners accelerated the development of F82H. Almost 30 years after, F82H is still used in several joint international collaborations. Another example is the joint irradiation programs. Irradiation facilities are becoming scarce and only available in a few countries. This type of collaboration is now extended to RAFM ODS steel developments. Since only a few companies are capable of fabricating these steels, without grouping the orders together at an international level to increase demand, companies will not invest in their productions.

Another example of the international collaboration is the work done on an intense neutron source (IFMIF). From the beginning it was well known that the irradiation experiments carried out in fission reactors are not representative of He/dpa ratio found in fusion reactors. A group attached to the Annex II of IEA fusion implementing agreement has been working for years on design and validation of an IFMIF. This work is now continuing under a Broader Approach agreement between EU and Japan. Associated with it is a user group for definition and qualification of small specimen technology. Trend in use of small specimens is inevitable: (a) because of the need to reduce the amount of irradiated materials, (b) to fit a large number of specimens in a small irradiation capsule.

6. Conclusions

Fusion materials community has been developing low activation ferritic/martensitic steels for more than 30 years. Two of these steels have reached maturity and are now finding their way in design codebooks. Others will follow soon.

Materials design properties of Eurofer have been added to RCC-MRx under A3.19AS in Section III, Tombe 1, Sub-Section Z. Initially design relevant allowables are derived for base metal and irradiation doses up to 3 dpa for the European TBMs. However, already data on base and weld metals are available at doses up to 80 dpa and the final goal is the full qualification of Eurofer steel for DEMO.

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