# Towards a Reduced Activation Structural Materials Database for Fusion DEMO Reactors

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Abstract. The development of First Wall, Blanket and Divertor materials which are capable of withstanding many years the high neutron and heat fluxes, is a critical path to fusion power. Therefore, the timely availability of a sound materials database has become an indispensable element in international fusion road maps. In order to provide materials design data for short term needs of ITER Test Blanket Modules and for a DEMOnstration fusion reactor, a wealth of R&D results on the European reduced activation ferritic-martensitic steel EUROFER, and on oxide dispersion strengthened variants are being characterized, mainly in the temperature window 250-650 °C. The characterisation includes irradiations up to 15 dpa in the mixed spectrum reactor HFR and up to 75 dpa in the fast breeder reactor BOR60. Industrial EUROFER-batches of 3.5 and 7.5 tons have been produced with a variety of semi-finished, quality-assured product forms. To increase thermal efficiency of blankets, high temperature resistant SiC<sub>f</sub>/SiC channel inserts for liquid metal coolant tubes are also developed. Regarding radiation damage resistance, a broad based reactor irradiation programs counts several steps from  $\leq$ 5dpa (ITER TBMs) up to 75 dpa (DEMO). For the European divertor designers, a materials data base is presently being set up for pure W and W alloys, and related reactor irradiations are foreseen with temperatures from 650 -1000 °C.

#### **1. Introduction**

The development of First Wall, Blanket and Divertor materials which are capable of withstanding the high neutron and heat fluxes for sufficiently high neutron fluence of at least 10-15 MWy/m<sup>2</sup>, corresponding to a component lifetime of at least five years, is a critical path to fusion power. As the construction of a Fusion Demonstration Reactor, DEMO, for demonstrating competitive electrical power generation will be the next important program milestone after ITER, the timely availability of a sound materials database has become an indispensable element in international fusion road maps. Even more, fast track scenarios like the Fast Track Experts Meeting convened by the president of the EU Research council and chaired by Prof. D. King [1], or the FESAC Fusion Development Path Panel chaired by Prof. R. Hazeltine [2] confirmed again that the material qualification and the related International Fusion Materials Irradiation Facility, IFMIF, are on a critical path. The fabrication technology and the materials database provided for design, construction, licensing and safe operation of a DEMO, must provide highly attractive properties, especially with respect to high thermal efficiency, availability, reliability, irradiation resistance and reduced activation capability.

After a brief review of strategies for materials development, this paper describes for the major blanket and divertor structural materials the present status of the fabrication and manufacturing technologies, and the achievements of the materials qualification, highlighting thereby examples of recent European progress.

### 2. Strategies for materials development

Supported by the "broader approach" discussions of the ITER negotiations, the major elements of formally developed [3] global fusion strategy scenarios have become very similar in the different parties. Figure 1 shows an example of an updated related road map [4]. The mentioned criteria have led to a worldwide concentration of the R&D efforts towards a few materials classes that are presently being developed in a largely coordinated manner under the umbrella of an IEA implementing agreement (ANNEX-II). For blanket applications, these are the structural materials classes of the RAFM steels (including nanodispersion strengthened variants), the vanadium alloys and the SiC fibre reinforced ceramic SiC composites. Although all three classes fulfil already today major requirements of reduced activation capability, the different materials are hardly comparable because the level of knowledge differs remarkably. The presently investigated vanadium alloys have an attractive combination of thermo-physical properties, high temperature strength and nuclear inventory decay, tend however after irradiation below about 420 °C to severe fracture toughness degradation and because of the pick-up of interstitial impurities (O, C, N) to pronounced embrittlement above about 550 °C. SiC/SiC composites feature attractive short and medium term nuclear inventories and high temperature strength. However, their structural integrity for high neutron doses must be fundamentally demonstrated, and besides pending structural design criteria the fabrication technology for large SiC/SiC components is not yet available. In view of the extreme heat load of helium gas cooled divertor concepts in some PPCS studies, the favoured materials are at present nanodispersion strengthened refractory alloys (e.g. W-La<sub>2</sub>O<sub>3</sub>) and W tiles at the plasma side, and high temperature resistant oxide dispersion strengthened (ODS) RAF steels at the backbone side.



FIG. 1: Cross-linking between the materials development and the fusion road map according to [4].

Reduced activation ferritic martensitic (RAFM) have by far the most advanced technological base of the three candidates and therefore became the reference as structural material for ITER test blanket modules (TBMs) and DEMO blankets in all parties:

- ITER: In order to get fully qualified TBMs assembled at the beginning of the operation phase I (around 2015), a detailed engineering design as well as fabrication and testing of TBMs is required already during 2006-2009 (Fig. 1). The ensuing activities (2010-2015) include fabrication, assembly and acceptance tests of final TBMs [5].
- DEMO: While the displacement damage level in ITER TBMs does not exceed about 3dpa (displacements per atoms) during the entire lifetime, 25-30 dpa will be achieved in

DEMO blankets within one year. That is, the neutron and thermo-mechanical loads of DEMO operating conditions are much more demanding. For DEMO design & licensing (fig. 1), all necessary materials should be qualified up to about 70-80 dpa in a fusion relevant neutron environment like IFMIF until around 2022.

Within the European Union, the two major breeder blanket concepts presently being developed are the Helium Cooled Pebble Bed (HCPB), and the Helium Cooled Lithium Lead (HCLL) blankets. For both concepts, different conceptual designs are being discussed with temperature windows in the range 250-550 °C for conservative approaches based on "reference" reduced activation ferritic-martensitic (RAFM) steels, and in the range 300-650 °C for more advanced versions, taking into account RAFM ODS steels. In addition, high thermal efficiencies beyond ~45% could be achieved by the use of SiC<sub>f</sub>/SiC channel inserts inside LiPb coolant tubes in a Dual Coolant concept. In order to timely provide a related materials database for the Test Blanket Modules of ITER and for DEMO, the European strategy for materials development is largely governed by the key issues (i) high neutron damage resistance, (ii) good mechanical properties at high operating temperature, and (iii) low activation properties. Similar approaches with different emphasis exist in Japan, the RF, and US.

## 3. Reduced activation steels of EUROFER type

### 3.1 Alloy selection, heat production, joining and manufacturing technologies

RAFM steels are modified compositions of conventional ferritic-martensitic 8-12% Cr-MoVNb steels mainly by exchanging Mo, Nb and Ni with W and Ta in order to obtain low activation capability [6]. To meet the ambitious time schedule, an integrated research and test program has been set up that includes besides broad based alloy development and qualification activities also various joining and manufacturing technologies of test modules and mockups. As a result of a systematic screening of many reduced activation ferritic-martensitic (RAFM) laboratory heats in Europe [7-10], and taking into account experience from similar programs in Japan and US, the 9% CrWVTa alloy EUROFER-97 was specified and industrial batches of 3.5 and 7.5 tons have been produced by two different EU companies with 10 different heats and 18 different semi-finished, quality-assured product forms (forged bars, plates, tubes wires) in various dimensions. Table 1 shows the chemical compositions of EUROFER-97 and the Japanese reference RAFM steel F82Hmod with upper limits of radiologically undesired elements. The produced RAFM steels meet already today the requirement of being low level waste after 80-100 years of DEMO reactor shutdown. The next step is the alloy fine tuning on the basis of substantial results from fission reactor irradiations and the subsequent definition and fabrication of EUROFER-II around end of 2006. An additional optimisation (EUROFER-III) aims at a substantial reduction of radiological impurities. This is considered to be technically possible on the expense of costs and would further significantly reduce the remaining  $\gamma$ -dose rate beyond 100 years, but would have no measurable impact on mechanical properties.

The main fabrication technologies presently examined for European breeder blanket structures include (i) bending, cutting and machining of different EUROFER plates, (ii) joining of complex shaped semi-finished products with many internal ducts, and (iii) heat treatments to ensure long-term aging resistance and to adjust a well defined microstructure that guarantees appropriate ductility and fracture toughness properties.

As joining technologies play a key role in the timely qualification of all breeder blankets, a broad based technology program has been launched in Europe on EUROFER-97 steel prod-

ucts with several associations involved. The joining technologies included until now (i) TIG welds, (ii) electron beam welds, (iii) laser welds, (iv) fusion welds under uni-axial pressure, as well as (v) solid-solid HIP joints and powder consolidated blocks. The welded product forms ranged from laboratory scale tensile and fracture toughness samples for a systematic analysis of post-weld heat treatments (PWHT) on mechanical and microstructural properties to medium-scale mock-up fabrication with structural integrity tests after PWHT. An overview on EUROFER-specific manufacturing technologies with special emphasis on results of joining techniques is given in [11]. Extensive chipless shaping and joining experience taking into account different welding procedures and powder technology product forms have demonstrated that EUROFER type steel complies with a wide range of established manufacturing processes.

	Radiologically	Eurofer 97 specified	Eurofer 97 achieved	F82Hmod.Heat 9741
	desired (ppm)	(mass-%)	(mass-%)	(mass-%)
A) C		0.09 - 0.12 [ 0.11 ]	0.11 - 0.12	0.09
Cr		8.5 - 9.5 [ 9.0 ]	8.82 - 8.96	7.7
W		1.0 - 1.2 [ 1.1 ]	1.07 - 1.15	1.94
Mn		0.20 - 0.60 [ 0.40 ]	0.38 - 0.49	0.16
V		0. 15 - 0.25	0.18 - 0.20	0.16
Та		0.10 - 0.14 [0.12]	0.13 - 0.15	0.02
$N_2$		0.015 - 0.045 [ 0.030 ]	0.018 - 0.034	0.006
Р		< 0.005	0.004 - 0.005	0.002
S		< 0.005	0.003 - 0.004	0.002
В		< 0.001	0.0005 - 0.0009	0.0002
O <sub>2</sub>		< 0.01	0.0013 - 0.0018	(0.01)
B) Nb	< 0.01 ppm	[< 0.001 (10 ppm)]	0.0002 - 0.0007	0.0001 (1 ppm)
Мо	< 1 ppm	[< 0.005 (50 ppm)]	0.001 - 0.0032	0.003 (30 ppm)
Ni	< 10 ppm	[< 0.005 (50 ppm)]	0.007 - (0.028)	0.02 (200 ppm)
Cu	< 10 ppm	[< 0.005 (50 ppm)]	0.0015 - (0.022)	0.01 (100 ppm)
Al	< 1 ppm	[< 0.01 (100 ppm)]	0.006 - 0.009	0.003 (30 ppm)
Ti	< 200 ppm	< 0.01 (100 ppm)	0.005 - 0.009	0.01 (100 ppm)
Si	< 400 ppm	<0.05 (500 ppm)	0.04 - 0.07	0.11 (1100 ppm)
Co	< 10 ppm	[< 0.005 (50 ppm)]	0.003 - 0.007	0.05 (50 ppm)

Table 1: Chemical composition of Eurofer 97 compared to F82Hmod; target values in [..].A) Main alloying elements, B) radiologically undesired elements in mass% and (ppm)

### 3. 2 Materials design limits and status of materials database

*Heat treatment, aging and physical properties:* Broad based characterizations and recommended heat treatments are available for the RAFM steels EUROFER-97 and F82Hmod. For EUROFER-97, the recommended two-stage heat treat treatment varies slightly depending on the product form: Normalization at 980-1040 °C for 27-30 min, followed by tempering at 750-760 °C for 90-120 min. All produced heats, semi-finished products and joints were post heat treated and analysed in various EU laboratories with emphasis on traceability and reproducibility. Until now, the in-depth chemical and homogeneity analyses have not revealed any abnormalities. The EUROFER-97 grain size is about 2-3 times lower with respect to F82Hmod. Continuous CCT diagrams have been analysed in detail together with the transformation, long-term aging, hardening and thermal expansion ( $\alpha_m$  and  $\alpha_i$ ) behaviour in the temperature range from RT-1120 °C [8]. Other physical properties include Elastic (E) and shear moduli (G), Poisson's ratio (v), electrical resistivity ( $\Omega$ ), Density ( $\rho$ ), specific heat ( $C_p$ ), thermal conductivity (k) and diffusivity (a), as well as magnetic properties.

*Tensile properties:* On the basis of diversified EU laboratories a reliable database exists on different unirradiated EUROFER-97 heats, product forms and orientations in the temperature

range RT-750 °C, both on base and welded samples. Tensile strength and ductility are almost unaffected by aging at 280-600 °C up to 3300 h. In the temperature range around 250-300 °C, which is most relevant for "low temperature irradiation embrittlement" mixed spectrum reactor irradiations exist up to about 9 dpa [12-14]. The related radiation induced strength increase of  $\Delta \sigma_y \cong 500$  MPa and the according total elongation of a few percent is within the scatter band in agreement with F82mod data.

Impact toughness and Fracture toughness: Impact tests have been carried out with standard and KLST-type specimens on all EUROFER-97 half products in all heat treated, aged and

joined conditions. The common KLST impact tests reveal in the asreceived condition a ductile to brittle transition temperature (DBTT) of about -90 °C (Fig. 2). After irradiation at 300 °C to 8 dpa this DBTT is increased to about 40 °C [12]. DBTT of solid-solid HIP joints is between - 60 and -90 °C, depending on the PWHT. Besides DBTT, upper shelf and lower shelf energies and FATT were determined.

Fracture toughness data based on CT, 1/2T CT and pre-cracked Charpy specimens have been measured in the heat treated condition in EU



FIG. 2: KLST impact transition curves for EUROFER-97 8 mm plate after 300 °C irradiation

laboratories. According to the master curve approach for CT specimens,  $T_0$  is equal to -30 °C. With respect to the international approach of qualifying miniaturized samples for fracture toughness, an overview is given in [15].

*Fatigue and creep fatigue:* For isothermal, strain controlled (creep) fatigue experiments complete datasets are available for EUROFER-97 in some EU laboratories from RT-550 °C up to a total strain range of  $\Delta \epsilon_t = 1.5$  %, and for dwell times in the compression and/or tensile phase from 0-600 s. The datasets include a sufficiently high number of stress-strain hysteresis loops per fatigue test and allows already a satisfactory correlation between  $\Delta \epsilon_t$  and the number of cycles to failure N<sub>f</sub> for a given set of machined specimens. However, for reliable lifetime prediction models, the impact of surface roughness on N<sub>f</sub> and a correlation between micro-crack initiation and propagation with N<sub>f</sub> needs still a lot more R&D work. Thermal creep fatigue with dwell times of 1000 s either in the compression or in the tensile phase, and with temperature windows from 100 to (500-550) °C reveals lifetimes N<sub>f</sub> between 400 and 9000 cycles, depending on the experimental conditions. However, N<sub>f</sub> of such thermally fatigued EURO-FER-97 is reduced by about one order of magnitude during alternating dwell times at high and low temperatures.

*Creep and creep rupture:* In the design relevant temperature and stress range broad based creep-rupture experiments between 450 and 600 °C up to 30 000 h creep rupture time have been executed on EUROFER-97 heats in two EU laboratories. Creep behaviour, stress exponents, 1% yield limits and rupture times showed no deviations from expected behaviour, confirming long term stability and predictability [8]. Emphasis is presently given on the safety relevant low-stress range (100 MPa and below).

*Fission reactor irradiations:* Besides already available results mainly from the mixed spectrum reactors BR2 and HFR in the lower dose regime, broad based HFR irradiations on 11 different heats have been performed between 60 and 450 °C with displacement damage levels up to 15 dpa, and in the fast breeder reactor BOR60 at 330 °C up to 30 dpa. The different heats include besides some reference RAFM steels various heats of EUROFER-97, EURO-FER-ODS, as well as <sup>10</sup>B and <sup>11</sup>B doped EUROFER to simulate helium effects. Significant PIE results will likely be available end of 2005. 75 dpa data after 330 °C irradiation will become available in about two years. Although irradiated EUROFER specimens have been examined at present only at 10 dpa and below, it can be stated that irradiation induced hardening, ductility reduction and fracture toughness degradation are highly superior to irradiated conventional ferritic-martensitic steels like EM10, HT9, MANET and T91. After irradiation, diffusion welded material performs worse than plate in the impact and fracture tests, but HIP welded material gives good impact results after irradiation.

*Materials design limit data, modelling and constitutive equations:* The materials community has to meet requests coming from "General Design Requirements Documents" or "ITER Interim Structural Design Criteria" in due time. Therefore, not only a sound database on materials properties is presently set up for EUROFER-97 but also data files containing traceable information on (i) procurement specification, (ii) grades of material used in the ITER TBM

relevant specifications, and (iii) section designations corresponding to material properties groups. These properties groups contain besides the analysed materials behaviour especially the "Material Design Limit Data". For EUROFER-97 such data are drafted in [16]. Besides the materials data a wealth of modelling activities is needed not only for a scientific understanding of properties but also for safety and availability assessments as well as for lifetime prediction of components. Finally, within the materials design interface activities constitutive equations need to be established further in strong interaction with fusion specific code developments.

*Materials database:* The data from the resulting R&D activities are compiled in relational data bases in Japan and Europe. An example of a EU database is given in Fig. 3, and further design relevant EUROFER-97 data are collected in [16].



FIG. 3: Example of data sheets from an European materials data base

### 3.3 Improvement of high temperature strength - the RAF(M) ODS steels

For specific blanket and divertor applications a replacement of presently considered conventionally produced RAFM steels by suitable ODS alloys allows a substantial increase of the operating temperature from ~550 °C to about 650 °C or even more. This has been shown by long-term ( $\leq 10,000$  h between 600 and 700 °C) creep rupture tests on specimens made of EUROFER97-ODS. A large material characterisation program, including reactor irradiation to 15, 30 and 70 dpa, is ongoing. A breakthrough has been achieved at FZK in overcoming

the poor high temperature ductility and ductile-to-brittle-transition temperature (DBTT) of first generation RAFM-ODS and commercial ferritic ODS alloys: the production route of the mechanically alloyed EUROFER-0.3wt%  $Y_2O_3$  included rolling in the so-called cross-roll technique with industrial partners, followed by appropriate thermal treatment. As a result, yield and ultimate tensile strength of the ODS-Eurofer are raised by 50% and more, compared to the non-ODS RAFM steels like Eurofer 97 and F82H mod. The total and uniform elongation of the ODS steel are superior over the whole temperature range to that of common RAFM steels. Impact tests on sub-size KLST specimens show greatly improved DBTT which could be shifted from +120°C for hipped ODS-Eurofer of the first generation to values well below 0°C. To establish reliable joining between ODS and RAFM steels, a diversified program on diffusion weldments was performed, showing that between RT and 700 °C all joints reached strength and ductility of the base EUROFER steel. Analytical High Resolution TEM has been used to explain the encouraging behaviour of the European RAFM-ODS batches.



Fig. 4: Larson-Miler plot for EUROFER-97 with different heat treatments in comparison with oxide dispersion strengthened EUROFER-ODS and F82Hmod (red broken line)

#### 3.4 SiC<sub>f</sub>/SiC composites and refractory alloys

SiC<sub>f</sub>/SiC composites are considered in the world as a most promising structural material option for a very high temperature operating window for blankets. Presently the results of neutron irradiation effects on SiC<sub>f</sub>/SiC composites up to 6 dpa are collected in Europe with irradiation temperatures of ~600 and 900 °C, respectively. Reductions in the bending modulus and dynamic modulus of some types of SiC<sub>f</sub>/SiC composite have been observed, where others remained unaffected. Although neutron radiation strongly reduces the thermal conductivity, the results indicate that with proper selection of manufacturing processes improvements can be realised. In the EU both 2-D and 3-D SiC<sub>f</sub>/SiC composites have been manufactured in close co-operation with Japan and the US to optimise the 3-D fibres architecture. Strength values of 200 MPa can be accomplished and the aim is for thermal conductivity over 20 W/mK.

In addition to the blanket activities, a modular He gas cooled divertor is being developed within the framework of the EU power plant conceptual study. As  $\sim 10 \text{ MW/m}^2$  and more needs to be removed, the design is presently based on tiles made of W ( $\sim 2000 \text{ °C}$ ), as well as

on structural materials like W-alloy (~700-1300 °C) and RAF(M)-ODS steel (~650°C). Also for such divertor designs, a suitable materials data base must be generated. The improvement and qualification of tungsten alloys as structural materials for high temperature divertor applications is challenging. Severe plastic deformation of pure W and W alloys improves ductility, but does not prevent from re-crystallisation between 850 and 1200°C. Tensile, aging and long-term creep rupture tests on commercially available W and W-1% La<sub>2</sub>O<sub>3</sub> are presently being done in the temperature window of interest, in order to get a preliminary database for divertor designers. In addition, R&D has started to improve the high temperature stability of W-alloys, and irradiation campaigns are being launched in Europe to get an initial database at two temperature levels between 650 °C and 1000 °C.

### Acknowledgement

Appreciation is given to all researchers of the European fusion materials development program, and besides EFDA and the EU commission also to the involved institutions and associations for their ongoing support.

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