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The development of EUROFER reduced activation steel

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Abstract

Ferritic martensitic steels show limited swelling and susceptibility to helium effects and can be made with low activation chemical compositions. These properties make them the reference steel for the development of breeding blankets in fusion power plants. EUROFER97 is the European implementation of such a steel, where experience gained from an IEA co-operation with Japan and the US is also implemented. Results obtained so far show that EUROFER steel has attractive mechanical properties even after long ageing times. Compatibility tests in water and PbLi17 are in progress. Oxidised aluminium is the most effective protective layer in PbLi17. The displacement damage and helium formation strongly influence the hydrogen transport in the steel. Present experiments should be backed by tests in a more fusion relevant environment, e.g. IFMIF. The 2.5 dpa neutron irradiations at low temperatures result in a higher DBTT. High dose irradiations, up to 80 dpa, are underway. The early results of ODS grades with EUROFER steel composition show potential of these grades for increasing the operating temperature with 100–150 K.

Keywords: EUROFER; Reduced activation steel; PbLi17

1. Introduction

Nature offers a limited amount of solutions for the near plasma combination of the major structural materials requirements: iron, vanadium, chromium, silicon and carbon [1].

The experience in fission power plants with conventional austenitic stainless steel up to high neutron fluences, showed as limitations helium embrittlement and swelling. The development of ferritic steels for fast reactor in-core components addressed the main austenitic steel drawbacks: helium embrittlement and swelling up to high, > 100 dpa, neutron exposures [2].

In the frame of an IEA implementing agreement, US, Japan, and EU co-operate to develop ferritic martensitic steels for fusion reactors that would keep the activation levels low and at the

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same time resist irradiation embrittlement, particularly at temperatures below 700 K. The work undertaken soon converged on 9Cr steels with small additions of tungsten, $<2.0\%$, vanadium, $<0.3\%$, and tantalum, $<0.2\%$. It also recognised that the impurity level of the steel must be well controlled in order to improve properties and keep the activation at an acceptable level. The OPTIFER steel developed at FZK is an example of this approach. In Japan, an industrial scale batch of such a reduced activation ferritic martensitic, RAFM steel, F82H, was produced and tested by the IEA partners [3]. The results paved the way for the production of a slightly modified industrial batch of RAFM steel in the EU, designated EUROFER97. This steel cast is presently being characterised in the EU in order to obtain design data for the blanket modules to be tested in International Thermonuclear Experimental Reactor (ITER) and to guide the way for further improvements.

2. Manufacturing

The fabrication of the EUROFER97 ingots used selected raw materials resulting in chemical compositions that did not satisfy the specification for some impurities (Nb, Ni, Cu, and Co). Impurity increases can be explained by pick-up from small residues left in equipment used for producing conventional steel compositions. Impurity levels could be reduced significantly by the use of dedicated steelmaking equipment.

The level of impurities EUROFER97 has reached, puts it in the class of Low Level Waste 100 years after service [4]. In order to move into the Hands-on Level domain the activation level has to be reduced by two orders of magnitude. This is technically feasible; it requires increased feedstock control and production lines reserved for low activation steel only.

At present priority is given to the quality of chemical analyses and development of low concentration calibration standards for such analyses. Paul et al. [5] give an example of a successful production route for a 1 ppm standard allowing the accurate measurement of uranium content in steel.

Powder production of EUROFER97 for mechanical alloying and hot isostatic pressing has been accomplished. Major concerns here are gas pick-up and contamination with activating impurities. HIP cycles have been successfully used for consolidating powder and joining solid parts [6,7]. Various fusion welding techniques have been applied, including inert gas, electron beam and laser beam welding [8]. Also dissimilar welds, RAFM and 316, have been made according to specification.

3. Mechanical properties

The 0.2% yield stress, YS, and ultimate tensile strength, UTS, of EUROFER97 measured up to over 1000 K, are similar to those of F82H and the OPTIFER steel. Spaetig [9] has developed a set of constitutive equations based on dislocation theory to provide the designers with an accurate tool for plasticity analyses.

The creep strengths of EUROFER97, measured in the range from 720 to 920 K by Schirra et al. [10], have similar trends as those of the other two RAFM steels.

The impact toughness energies recorded for EUROFER97 show a ductile to brittle transition, DBTT, around 200 K, whereas OPTIFER shows DBTTs dependent on their grain size in the range of 185–215 K. The slightly higher DBTT of F82H at 230 K is attributed to the larger grain size and the higher oxygen content as compared to those of EUROFER97.

Rensman has compared the fracture toughness properties of EUROFER97 with those of F82H [11]. The first shows a sharp transition at 180 K, whereas the latter shows a scatter in transition, but still below 180 K. The impact toughness tests produce different transition temperatures than the fracture toughness tests, which should be reflected in the design limits.

Riesch-Oppermann [12] investigated the local approach to be applied for the design with RAFM steels. He considers the approach as complementary to the master curve method. Results of

EUROFER97 notched tensile specimens corroborate the analyses made in the spirit of the R6 Code [13].

Petersen and coworkers [14] have investigated the thermal and iso-thermal fatigue properties of EUROFER97 at the temperatures up to 873 K, Fig. 1. The iso-thermal low cycle fatigue shows softening after a few cycles up to failure that is hardly dependent on test temperature up to 600 K. The strain amplitude has no influence of the magnitude of the softening. Over that temperature the softening increases rapidly by reduction of the internal stresses exemplified by the hysteresis loops. The softening is brought about by the evolution of a hard lath structure into a softer dislocation cell structure.

4. Microstructural stability

Besides the chemical composition and working production route the heat treatments of the steel prior to application determine the final properties of RAFM steels such as EUROFER97. The austenitisation temperatures of these steels are in the range of 1250–1325 K, the tempering temperatures amount from 950 to 1025 K, Fig. 2.

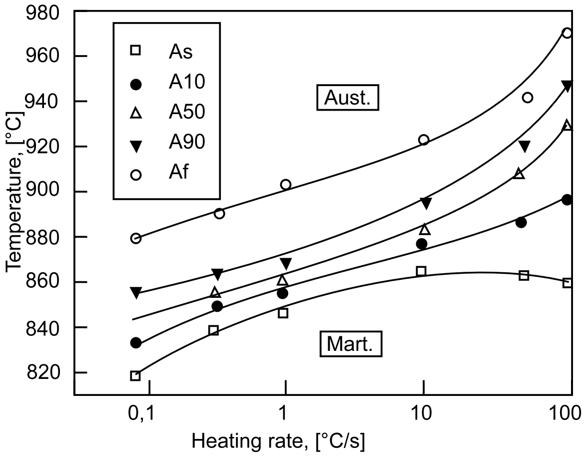

Fig. 2. On-heating transformation temperatures of EUROFER97 steel from Ref. [15].

Danon [15] observed that the prior austenitising grains are stable up to 1325 K. Above that temperature grain growth is observed. EUROFER is heat-treated in a manner to obtain a fully martensitic microstructure that should remain stable during service.

CCT diagrams with cooling rates in the range of 15–300 K s^{-1} obtained for EUROFER are similar to other RAFM steels. This means that a wide range of EUROFER component thicknesses can be heat-treated.

The ITER test blanket modules have estimated typical maximum thermal service conditions of 20 000 h at 823 K. It is therefore necessary to quantify the potential ageing susceptibility of the RAFM steel. Fernandez et al. [16] measured the DBTT values and tensile properties after 5000 h ageing at 773 K. They did not observe a significant change compared to the impact toughness values and tensile data measured for the as-received condition. Schirra et al. and Alamo [10,17] report that equivalent ageing treatments of 1050 h at 875 K or 3330 h at 855 K do not affect the tensile properties significantly, Fig. 3a and b. Ageing treatments at much higher temperatures decrease the tensile strength. The ITER test blanket modules thermal service conditions are not a concern for EUROFER97.

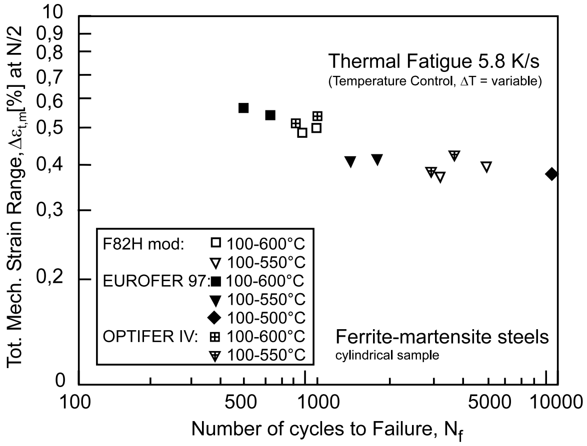

Fig. 1. Thermal fatigue endurance versus total mechanical strain range for EUROFER97 and other RAFM steels from Ref. [14].

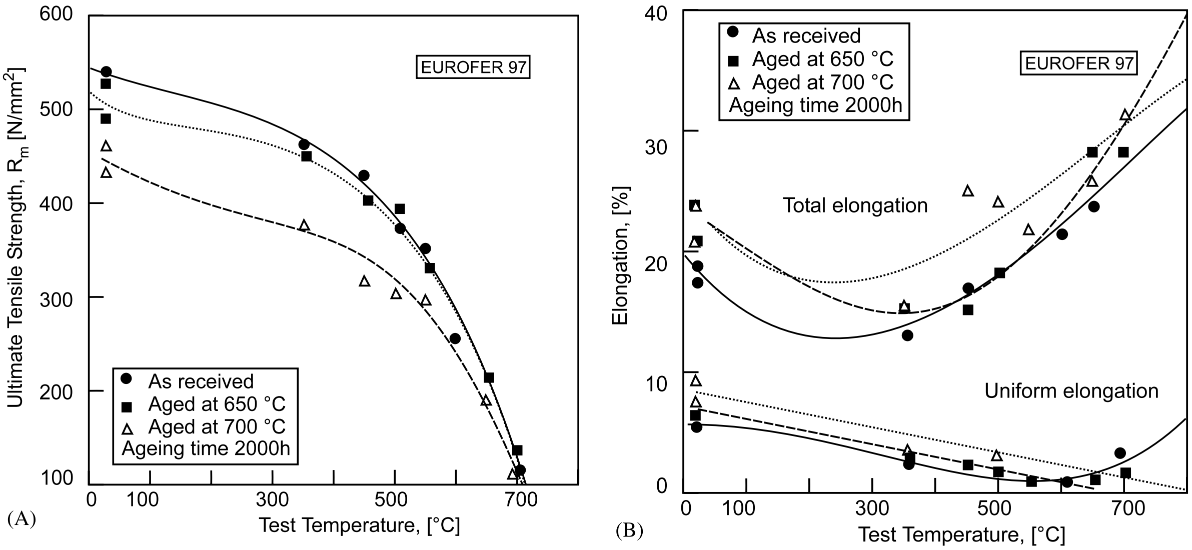

Fig. 3. (A) Effects of thermal ageing on the UTS measured at different test temperatures, from Ref. [17]. (B) Effects of thermal ageing on the tensile elongation measured at different test temperatures, from Ref. [17].

5. Radiation effects

The program in the EU proceeds with irradiations of EUROFER97 to a wide range of radiation damage, starting from 0.3 to 1.0, 2.5, 8, 15, 30 and 80 dpa. The steel irradiated to the lower damage levels supports the study of the build-up of radiation damage and the theoretical explanation. The highest dose levels provide steels with damage relevant for the levels envisaged for the blanket structural application.

The BOR-60 in Dimitrovgrad provides the higher doses for hundreds of test specimens [18]. This fast reactor is sodium cooled and hence the minimum irradiation temperature is about 610 K minimally. The lower dose levels are provided by materials test reactors such as the BR2, R2 and HFR that allow minimum temperatures down to 330 K.

The post-irradiation testing of EUROFER97 has started obviously with the lower doses. Rensman [19] reports tensile test results after a dose level up to 2.5 dpa showing 0.2% yield stresses and ultimate tensile strengths comparable with those measured for F82H for example. The transition fracture toughness has been plotted in Fig. 4 for EUROFER97, F82H and a laboratory scale heat.

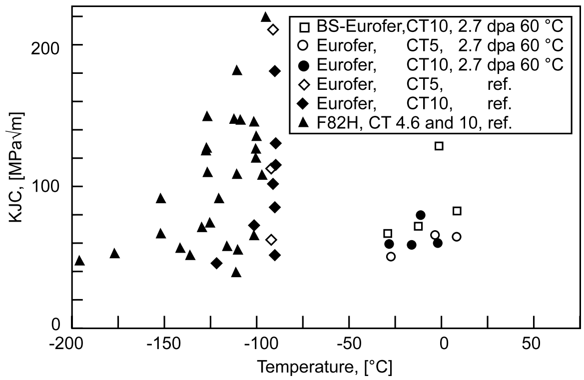

Fig. 4. Transition fracture toughness values for EUROFER97 and other RAFM steels in reference and irradiated conditions, from Ref. [19].

The effect of irradiation on the toughness on EUROFER97 and F82H is quite similar. Some experimental RAFM steel batches show that toughness reductions by irradiation can be limited through heat treatments and better impurity control.

Preininger [20] analysed the effect of helium on tensile ductility and impact energy. Helium influences the precipitate behaviour under mechanical loading and thus the ductility.

Both in the HFR and BOR60 steel samples with additions of natural boron up to 82 wppm and boron10 up to 1160 wppm are in progress to simulate service conditions. The limitations of this approach have been brought forward by Klueh et al. [21].

IFMIF will allow simulations with 14 MeV neutrons much closer to the power plant environment and they are decisive in the accurate quantification and explanation of the combined effects of helium and displacement damage.

6. Compatibility

Raquet [22] reports that the corrosion and stress corrosion of EUROFER97 in a hydrogenized aqueous environment is comparable to that of F82H and similar RAFM steels.

Maday [23] found that the coolant chemistry controls the load controlled fatigue at 510 K strongly. The experiments conducted on RAFM steels of the 8Cr2WVTa class showed that the normalised and tempered condition shows ductile features on fractures.

EUROFER97 corrosion and tensile specimens were exposed to flowing Pb17Li. The test temperature was 753 K and the flow rate of the liquid metal was 10^{-2} m s^{-1}. Tensile and corrosion specimens were taken out every 1500 h up to 4500 h. Fazio, [24], reports the steel exhibited a linear increase of the weight loss with time, resulting in about 40 μm per year. The liquid metal dissolves the steel elements with the formation of a Cr depleted layer, the detachment of which is enhanced by the liquid metal. The tensile tests performed at 753 K on the corroded specimens up to 4500 h reveal only slight effects on the strength and the area reduction of the steel. Glasbrenner [25] investigated the corrosion behaviour of aluminised coatings at 753 K in flowing Pb17Li (0.3 m s^{-1}). This work has shown that coatings having an α-Al_2O_3 scale due to high temperature treatment suppress the liquid metal attack effectively up to 10 000 h. The coating having a γ-Al_2O_3 phase, due to low temperature processes, exhibited liquid metal corrosion.

The equation which expresses the hydrogen permeability (Φ) as a function of temperature on EUROFER97 steel is reported by Aiello [26] as $\Phi = 1.53 \times 10^{-8} e^{-38\,280/RT}$ (mol m^{-1} s^{-1} $Pa^{-1/2}$), which is significantly lower than for the F82H steel. In addition Aiello [26] reported the threshold hydrogen embrittlement concentration, measured on smooth specimens at room temperature, to be 1.8 wppm. Jung [27] has reported the effect of radiation damage to 0.01 dpa and injected helium to 1000 appm on desorption of deuterium from EUROFER97. The deuterium was thermally loaded or implanted with a beam from a cyclotron into samples of different thicknesses.

Loss of deuterium through thermal cycling in vacuum was measured. Analyses of the resulting peaks leads to the conclusion that a trap model with saturation, derived from permeation experiments, cannot describe the desorption behaviour in as-received condition.

Displacement damage and or implanted helium strongly retain the deuterium in the EUROFER97 samples.

7. High performance steels

The Oxide Dispersion Strengthening, ODS, of EUROFER97 is in the laboratory scale phase. Several European industries have atomised batches of the original EUROFER97 to powders. Consolidation of the EUROFER97 powder is done by HIP and extrusion after mechanical alloying with different oxides, but mostly with Y2O3. The resulting products have a ferritic microstructure, which has shown to be fairly stable according to Cayron [28].

The consolidated products have yield, and tensile strengths roughly 35% higher than plain EUROFER97 up to 900 K as shown by Lindau [29] and Alamo [30]. The ductility, in particular from extruded products is even higher than that of the base material.

The main concerns about RAFM steels remain their toughness after accumulation of large amounts, 100 dpa, of displacement damage and high, $\gg 1000$ appm helium and hydrogen. These essential factors are studied, but in isolation. As the effects of hydrogen already show the interaction of radiation damage, helium clusters and deuterium determine the mobility of the latter. A more representative test environment is thus mandatory. A 14 MeV source such as the International Fusion Material Irradiation Facility, IFMIF, is necessary to support reliable predictions about the RAFM behaviour in power plant blanket service.

In parallel modelling efforts in the EU are devoted to the interaction of displacement damage and transmutation products. The experimental results will support the validity of the models.

8. Conclusion

EUROFER97 is a representative of reduced activation ferritic martensitic, RAFM steel, manufactured on an industrial scale. The potential of further reduction of impurities and therefore promoting lower activation exists. Manufacturing technology of the steel has been demonstrated to have a broad basis. The properties of the steel meet the requirements of the test blanket module of ITER. The mechanical and compatibility properties are adequate, also in the neutron flux exposures expected in ITER. The potential of application in blankets for a demonstration fusion power plant is there, but experimental verification under conditions representative for the near plasma environment remains essential.

A test device such as IFMIF is mandatory to study the interaction of such effects as displacement damage, and hydrogen and helium formation on the steel toughness more realistically.

The tough ODS steel high temperature development has to be accompanied by manufacturing developments, which are crucial for the application in blanket structures.

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