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Journal of Nuclear Materials 307–311 (2002) 769–772

journal of nuclear materials

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Mechanical and microstructural properties of a hipped RAFM ODS-steel

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Abstract

Oxide dispersion strengthened (ODS) materials for structural applications in future fusion power reactors would allow to increase the operating temperature to approximately 650 °C. Two ODS variants of the 9%CrWVTa-RAFM steel EUROFER 97 with Y_2O_3 contents of 0.3 and 0.5 wt% have been produced. The microstructure of both powder and compacted material has been characterised by means of optical and electron microscopy (SEM, TEM). Tensile tests performed between RT and 850 °C show an increase of yield strength and ultimate tensile strength by 50% and more compared to the basic steel. The ductility values are sufficiently high. Creep tests between 600 and 700 °C up to test times of 5000 h confirm the superior creep strength of ODS-EUROFER. Impact tests reveal a similar impact behaviour to comparable ODS alloys, but the ductile-to-brittle transition temperature is substantially higher than that of EUROFER 97. Preliminary results of low-cycle fatigue show a promising fatigue behaviour of ODS-EUROFER.

1. Introduction

The efficiency of future fusion power plants is strongly dependent on the operating temperature. Conventional RAFM steels presently considered for structural applications limit the operating temperature to around 550 °C. Oxide dispersion strengthened (ODS) materials would allow an increase in the operating temperature of about 100 K. The benefit of ferritic ODS-alloys as cladding material for liquid metal fast breeder reactors was recognised in the late sixties [1]. ODS alloys have been attracting attention again recently and have been proposed also for fusion reactor application [2–6]. As a first step in developing a reduced-activation ferritic–martensitic ODS steel, an existing 9%CrWVTa-RAFM steel called EUROFER 97 [6–9] was chosen as base material and two variants with Y_2O_3 contents of 0.3 and 0.5 wt% have been produced by experienced industrial manufacturers. The scope of the presented work is the characterisation of the microstructure and mechanical properties of these alloys.

2. Experimental

The European RAFM steel EUROFER 97 with a basic composition of about 8.9 wt% Cr, 1.1 wt% W, 0.2 wt% V, 0.14 wt% Ta, 0.42 wt% Mn, 0.06 wt% Si, 0.11 wt% C and Fe for the balance was chosen for the production of two variants of ODS steels with different Y_2O_3 contents (0.3 and 0.5 wt%). Activation calculations showed that long-term activation is not impaired by the addition of yttria. The production process included inert gas atomisation of EUROFER (H.C. Starck) and subsequent mechanical alloying in industrial ball mills of attritor type by Plansee. Hot isostatic pressing was chosen as the appropriate consolidation process for the production of four bars of each heat 60 mm in diameter and 300 mm in length. Part of the material was made available to other European laboratories [10,14]. The hipping process is regarded as the most promising production route for nearly end-shaped

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PII: S0022-3115(02)01045-0

structures for future fusion reactors. This production process has the additional advantage that no anisotropy is induced as in the case of hot forming processes.

The microstructure of both powder and compacted materials has been characterised by means of optical and electron microscopy. A Philips environmental scanning electron microscope (ESEM) and high resolution TEM Philips CM 30 both furnished with energy dispersive X-ray analysers were used for the electron microscope examinations.

Miniaturised specimens were used for the determination of the mechanical behaviour. Small specimens test technology (SSTT) specimens [11] with 2 mm diameter and 7.6 mm gauge length were used for the tensile and low-cycle fatigue (LCF) tests and usual subsize KLST (Kleinstprobe acc. DIN 50115) specimens with $4 \times 4 \times 27$ mm^3, 1 mm notch depth, 0.1 mm notch root radius and 60° notch angle were used for the impact tests. Comparative tensile tests and the creep tests were performed using a specimen with 3 mm diameter and 18 mm gauge length. The specimen orientation for all tests was longitudinal to the rod axis. The tensile and LCF tests on SSTT specimens were performed using a Zwick Z030 universal testing machine in a vacuum of 8×10^{-7} mbar. The comparative tensile and the creep tests were performed under normal atmosphere. The applied strain rate for the tensile tests was 22×10^{-5} s^{-1} for SSTT and 17×10^{-5} s^{-1} for the larger specimens. The strain of the SSTT specimens was measured by remote-controlled extensometers that have been specially designed for the use in hot cells. The strain of the larger specimens, tested under air in an Instron 4505 machine, was measured by cross-head displacement. All mechanical tests were performed on material in the as-hipped condition.

3. Results and discussion

3.1. Microstructural examinations

The microstructure of the different powders and consolidated materials in different heat treatments has been characterised using optical microscopy, SEM, TEM and X-ray diffraction. With a few exceptions, the hipped material exhibits a quite homogenous equi-axed grain structure with a grain size of 2–8 μm. The boundaries of these grains are decorated with Cr-rich precipitates of $M_{23}C_6$ type. The matrix structure is ferritic in contrast to the basic EUROFER 97 steel that shows the typical lath structure of a tempered martensite with a prior austenite grain size between 11 and 23 μm [7] for austenitising temperatures up to 1040 °C. Hardening experiments showed that a martensitic transformation of ODS-EUROFER can only be obtained by rapid quenching in water, which cannot be regarded as appropriate for the heat treatment of large complicated blanket structures. One possible reason for this unexpected behaviour could be the loss of carbon during the atomisation process of EUROFER from 0.114 wt% for the bar material to 0.079 wt% for the powder. The nearby grain boundaries of the very small grains and a high density of yttria particles may act as sinks for the carbon and may thus retard the austenite–martensite transformation. The spatial distribution of the Y_2O_3 dispersoids, which are primarily responsible for the strengthening of ODS-EUROFER, is quite homogeneous. Nevertheless a variation of the local density of ODS particles by a factor of approximately 3 between different grains could be found. The observed particle size ranges from 4 to 30 nm with a mean size of 12 nm. Regions with a noticeable finer dispersion of yttria particles (2–10 nm, mean size 4 nm) could also be found. While the larger particles are incoherent to the matrix, a clear crystallographic orientation relationship between the 4–6 nm large particles could be found. The indicated planes in Fig. 1 correspond to type {2 2 2} planes of the cubic Y_2O_3 modification with a lattice constant of 1.06 nm. A publication of a more detailed analysis is in preparation.

3.2. Tensile tests

Miniaturised specimens 2 mm in diameter and a gauge length of 7.6 mm were fabricated from the as-received bars and subjected to tensile tests in the temperature range between RT and 850 °C. Comparative tests have also been performed on specimens 3 mm in diameter and 18 mm gauge length. Fig. 2 gives the ultimate tensile strength R_m and total elongation A of the two ODS alloys in comparison to the corresponding data of the basic steel EUROFER 97, which is similar to comparable RAFM steels like F82H mod [7–9]. The ultimate tensile strength of the ODS-EUROFER (0.5

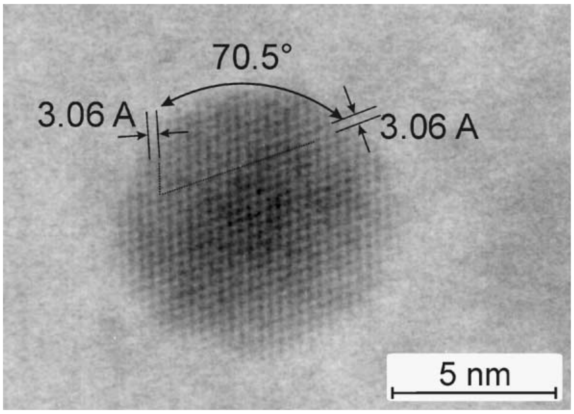

Fig. 1. High-resolution-TEM bright field image of an Y_2O_3 particle.

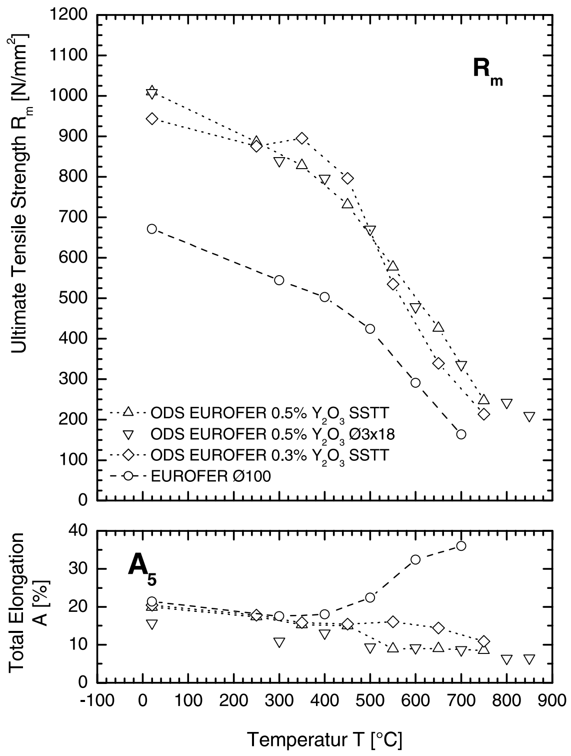

Fig. 2. Ultimate tensile strength and total elongation of different ODS alloys compared to EUROFER.

wt% Y_2O_3) is over 50% higher than of non-ODS RAFM steels. This gain in strength persisted at elevated temperatures. The data points of the SSTT and 3 mm specimens (upright, downward triangles) are arranged like a bead showing equivalence of data generated by small-scale specimens. The 0.3 wt% Y_2O_3 alloy has a slightly higher ultimate tensile strength in the temperature region between 350 and 450 °C, whereas it is slightly lower above 500 °C. The yield strength of ODS-EUROFER, not shown here, results in the same increase in strength compared to the base steel, but the alloy with the lower content of Y_2O_3 is always less or equal in strength compared to that with the higher content. The total elongation of the ODS alloys above 400 °C is lower than that of the non-ODS RAFM steels, but remains always above 6%, that seems to be sufficiently high. The uniform elongation of the ODS alloys with minimum values above 3% is always notedly higher than for the base steel.

3.3. Creep tests

The first uniaxial creep tests have been conducted at temperatures between 600 and 700 °C in air reaching rupture times up to 5000 h. The results are given in the Larson–Miller–Plot in Fig. 3 and are compared to the results of the RAFM steels EUROFER 97, OPTIFER, a precursor developmental alloy, and the Japanese RAFM reference steel F82H mod. ODS-EUROFER, containing 0.5 wt% Y_2O_3, shows a pronounced higher creep strength than the basic steel EUROFER 97 and comparable RAFM steels. The envisaged goal, to extend the operation temperature by 100 K, has been impressively fulfilled. ODS-EUROFER shows the same creep strength as EUROFER 97 but at about 100 K higher temperature.

3.4. Impact bending tests

Sub-size Charpy specimens of KLST type have been machined from one rod of the 0.3 wt% Y_2O_3 containing

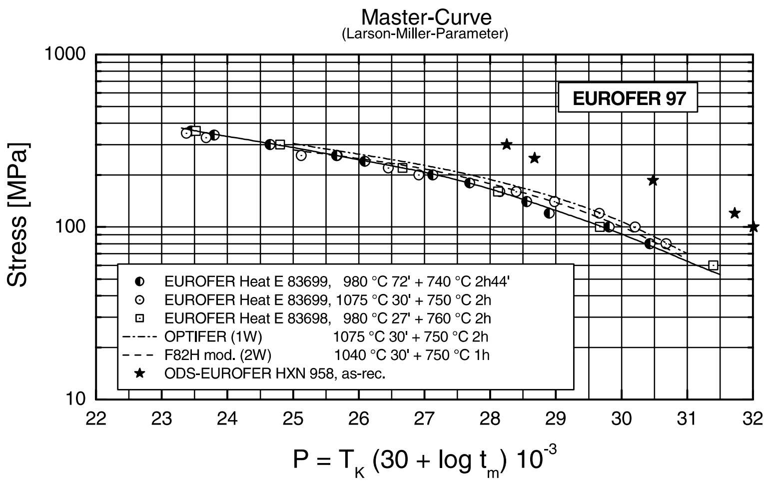

Fig. 3. Creep strength of ODS-EUROFER (0.5 wt% Y_2O_3) in comparison with RAFM steels EUROFER 97, OPTIFER and F82 H mod.

material and were tested in a fully instrumented 15 J pendulum machine. Fig. 4 shows the temperature dependence of the total absorbed energy of specimens fabricated from two different rods of ODS-EUROFER in comparison to EUROFER 97 [12–14]. The upper shelf energy, i.e. the maximum absorbed energy of the ODS alloy reaches values of about 5.4 J and is reduced by approximately 40% compared to the values of EUROFER 97, which reach roughly 9 J. Much more eye-catching is the shift of the ductile-to-brittle transition-temperature (DBTT) from −100 °C for EUROFER to values between 70 and 100 °C for the longitudinal specimens of ODS-EUROFER taken from two different rods of one heat. The data obtained by Lucon, SCK–CEN [14] show a slightly larger scatter than the FZK data and tend also to a slightly lower DBTT. However, it should be kept in mind that the fitted curves for EUROFER include also some scatter in the same order of magnitude. To estimate whether the observed deterioration of the impact properties of the examined ODS alloy is material-inherent or production related, is difficult since no comparable data of powder-hipped EUROFER are available at the moment. It was demonstrated in [15] that improvements are possible where impact data of a ferritic developmental and a commercial alloy (MA957) showed transition temperatures well below (−43 °C) and around room temperature, respectively. Unfortunately these data are not directly comparable since they were generated with sub-miniature specimens with a 1.5×1.5 mm^2 cross-section and 0.3 mm notch depth which pretend a lower DBTT than larger specimens. The improvement of the impact properties will be the subject of forthcoming investigations.

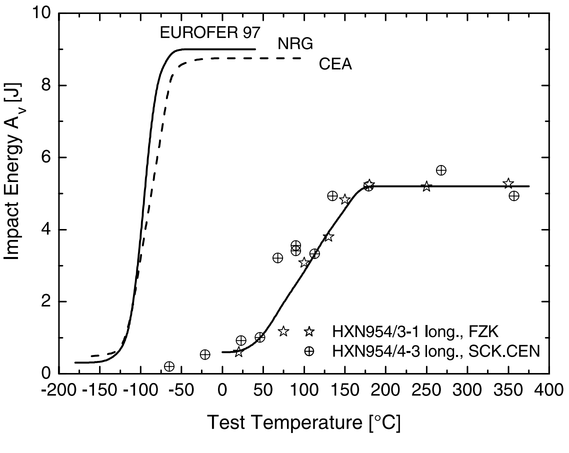

Fig. 4. Test temperature dependence of total absorbed energy of ODS-EUROFER (0.3 wt% Y_2O_3) in comparison with RAFM steel EUROFER 97.

3.5. Low-cycle fatigue tests

Preliminary results of strain-controlled isothermal LCF tests on the same type of specimens as used for the tensile tests [11] show the good LCF-behaviour of ODS-EUROFER. Compared to F82H mod, no cyclic softening, a lower plastic deformation, a substantially higher stress amplitude at a given strain and a higher lifetime can be observed at 250 °C. These experiments will be continued and extended to much higher temperatures.

4. Conclusions

It has been successfully demonstrated that it is possible to expand the temperature range for the application of RAFM steels by reinforcement with yttria. Two ODS-alloys based on EUROFER 97 with different yttria contents (0.3 and 0.5 wt%) have shown promising mechanical strength and sufficient ductility. The impact behaviour is not satisfying at present. The work on the mechanical behaviour (tensile, creep, impact, LCF) will be continued. The transformation behaviour of the ODS alloys deviates from conventional RAFM steels with similar composition. Nevertheless excellent properties (except for the impact properties) were achieved so that martensite formation, which is a pre-requisite for hardening and tempering treatment, might not be necessary to achieve the desired properties for applications up to 650 °C. Further studies will be necessary to understand the mechanisms involved.

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