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Assessment of ODS-14%Cr ferritic alloy for high temperature applications

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Abstract

The present paper evaluates the mechanical behaviour of the ODS-ferritic alloy, Fe–14Cr–1Ti–0.25Y_2O_3 (MA957), produced with two microstructures characterized by different grain sizes and degree of crystallographic texture. Tensile, creep and impact properties were found to be strongly dependent on the metallurgical condition. The neutron irradiation behaviour was also investigated. For this purpose, irradiation experiments were performed at 325 °C for various dose levels. The specimens of ODS-ferritic steel irradiated up to 6 dpa exhibited a lower irradiation-induced hardening and higher ductility values compared to conventional martensitic steels irradiated in the same conditions. The occurrence of α' precipitation was detected using small angle neutron scattering techniques.

1. Introduction

Oxide dispersion strengthened (ODS) alloys based on a Fe–Cr ferritic or martensitic matrix, offer the potential of application to higher operating temperatures ($T > 550$ °C) in fusion reactors, where conventional reduced activation ferritic–martensitic (RAFM) steels cannot be used any longer because of their low creep resistance [1,2].

ODS Fe-based alloys are the only materials presenting jointly two important properties: (a) a high dimensional stability under irradiation, i.e. high resistance to swelling and irradiation creep, due to their bcc structure, and (b) a potential high temperature strength resulting from the homogeneous dispersion of nanometric oxide particles.

ODS ferritic steels with a high chromium content (Cr > 12%) present a fully ferritic matrix and are envisaged for applications in an extended range of temperature. However, the level of high temperature strength is determined by several parameters like oxide distribution, grain size and texture. In the case of ODS-ferritic alloys, the main difficulty could be the anisotropy in mechanical properties resulting from the anisotropy of microstructure developed during fabrication.

The main objective of the present work is to summarize the mechanical behaviour of the ODS-14%Cr ferritic (MA957) alloy obtained with two typical microstructures characterized by a very fine and elongated grain microstructure (mean diameter 0.5 μm) and a recrystallized grain structure with a 10–50 μm diameter. The susceptibility to hardening and embrittlement induced by neutron irradiation at 325 °C for several dose levels was also investigated. The first results obtained up to 6 dpa are presented as well.

2. Experimental

The ODS-14%Cr (MA957) alloy was supplied as hot-extruded bars of 25 mm diameter with a nominal chemical composition (wt%) Fe–14Cr–1Ti–0.3Mo–0.25Y_2O_3. Subsequent working processes were applied in our laboratory.

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Tensile tests were performed using cylindrical specimens of 2 mm diameter and 12 mm gauge length with a strain rate of 7×10^{-4} s^{-1}. Specimens for creep tests were of 4 mm diameter and 20 mm gauge length.

Full impact energy curves were determined using Charpy-V sub-size specimens of 3 mm × 4 mm × 27 mm obtained along the axial direction of bars. The Ductile–Brittle transition temperature (DBTT) was deduced from the half-value of the upper shelf energy (USE) level.

Neutron irradiations were performed in the Osiris reactor (CEA) at 325 °C for several dose levels as described in [3].

Microstructural investigations were carried out at Leon Brillouin lab. (CEA) using small angle neutron scattering (SANS) techniques. The experimental conditions and data treatment are described elsewhere [4].

3. Microstructure

Due to the chemical composition, the ODS-14%Cr alloy is constituted of a fully ferritic matrix with a homogeneous dispersion of oxide particles of nanometer size.

This ODS-ferritic alloy presents a very fine microstructure after hot-extrusion and cold-deformation characterized by grains with a strong anisotropic shape of 500 nm diameter and a length/diameter ratio of 10–20. Other typical features of this microstructure is related to the high index of the deformation texture [5].

Recrystallization temperatures and recrystallized grain morphologies of ODS alloys strongly depend on the deformation process used and the amount of cold-work [6,7]. During recrystallization treatments, these types of materials could develop coarse grains of millimeter size. Nevertheless, a well controlled fabrication route including a cold-drawing process was established, which led to a recrystallized microstructure with intermediate grain sizes of 10–50 μm diameter as shown in Fig. 1. This cold-drawn/recrystallized condition is characterized by a minimum texture level that could be obtained for this type of ODS ferritic alloys [5,6].

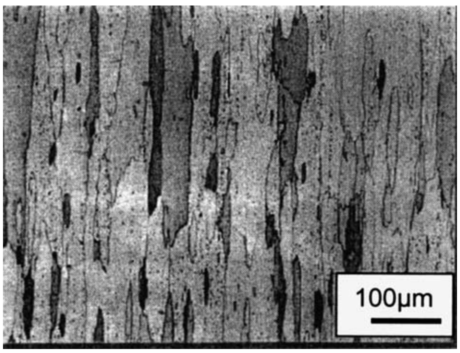

Fig. 1. Morphology of MA957 recrystallized grains obtained after deformation by cold-drawing and recrystallization heat-treatment at 1100 °C.

In the present work both types of microstructures, i.e., fine grain structure and cold-drawn/recrystallized condition are investigated. Both microstructures present the same distribution of oxide particle size according to results obtained by TEM and SANS. The average size of particles is about 12 nm and the distribution is extended from 1 to 25–30 nm.

4. Tensile properties

Fig. 2 compares the typical values of 0.2% proof stress, total elongation and reduction in area at rupture as a function of the test temperature for MA957 specimens having various conditions, that is, after hot-

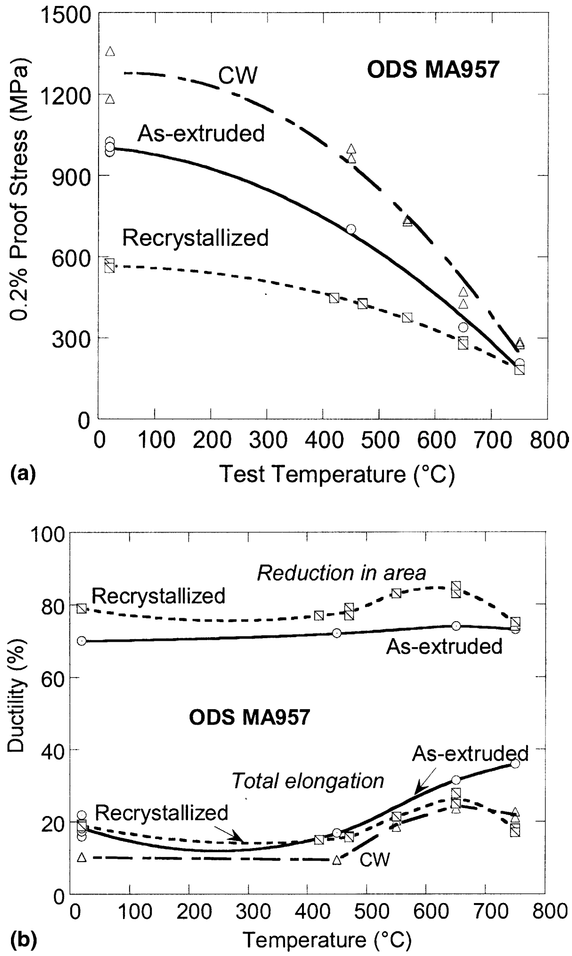

Fig. 2. Effects of the metallurgical condition on the tensile properties of MA957 alloy: (a) tensile strength; (b) ductility as a function of the test temperature. As-extruded and CW specimens presented very fine grain structures.

extrusion, extrusion followed by cold-work, and finally after recrystallization treatment.

As shown in Fig. 2(a), the tensile strength varies strongly with the microstructure and metallurgical condition. The recrystallized state is characterized by lower values of yield stress, especially at temperatures below 600 °C. Nevertheless, it is shown that recrystallization treatments enhanced the ductility, in particular the values of reduction in area to rupture, which is one of the main concerns in the ODS alloy development.

To estimate the anisotropy of tensile properties, some tests were performed in the transverse direction, that is perpendicular to the axial direction of bars, using small ring specimens [8]. In the case of fine grain bars, the tensile strength could be up to 30% lower than that measured in the axial direction. A very small difference was found for recrystallized specimens in agreement with the low texture intensity measured.

5. Impact properties

Full energy transition curves determined for the as-extruded and recrystallized specimens of MA957 alloy are shown in Fig. 3. The main difference between both conditions is given by the DBTT values, the fine grain microstructure presenting a very attractive value (−110 °C) compared to the recrystallized state (+60 °C). However, the recrystallized condition exhibits higher values of USE level.

6. Creep properties

The influence of the applied stress on the rupture time, determined at 650 and 700 °C for both types of microstructure, is presented in Fig. 4. As expected from tensile properties, the recrystallized specimens are less creep resistant than the samples having a fine grain structure for short rupture times, but their behaviour becomes comparable at 650 °C for times of about 10^4 h.

Results reported in Fig. 4 were obtained for specimens taken along the axial direction of bars. Due to the strong texture of the fine grain microstructure, a lower creep strength is expected for specimens oriented in the radial direction as predicted by the tensile behaviour. In contrast, nearly isotropic creep strength is expected along the radial and axial directions for recrystallized specimens.

For rupture times longer than 10^3 h, the ODS-14%Cr displays a higher creep resistance than the conventional (non ODS) ferritic–martensitic 9Cr2MoVNb (EM12) and Ti-stabilized austenitic steel 15Cr–15Ni (15/15Ti).

A typical feature of ODS creep behaviour is related to the high strain rate sensitivity to the applied stress [9–11]. This fact is shown by the high values of the stress exponent 'n' of the Norton creep law relating the strain rate $\dot{\varepsilon}$ and the applied stress σ, i.e., $\dot{\varepsilon} = AD_{\mathrm{v}}(\sigma/E)^n$ (where A is a material parameter, D_{v} the bulk diffusion coefficient, E the elastic modulus). In the case of recrystallized MA957 the stress exponent n is ∼50, much higher than values usually found for pure metals and conventional alloys (3–7). This particular creep behaviour is generally explained by the existence of a creep threshold stress σ_{th} below which the creep rate is negligible. From plots of the normalized strain rate $\dot{\varepsilon}/D_{\mathrm{v}}$ against the normalized

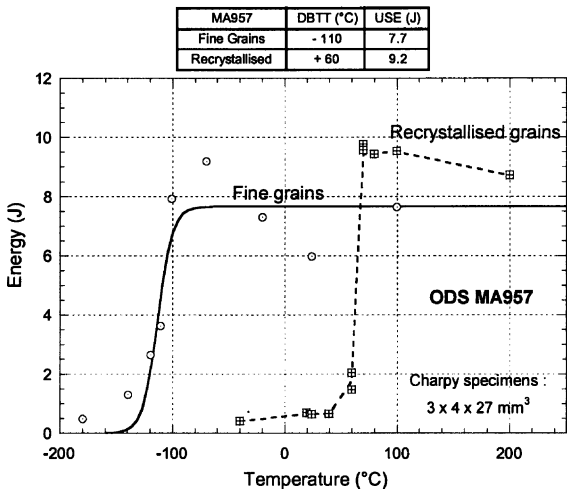

Fig. 3. Effects of the microstructure on the impact properties of MA957 ODS-alloy.

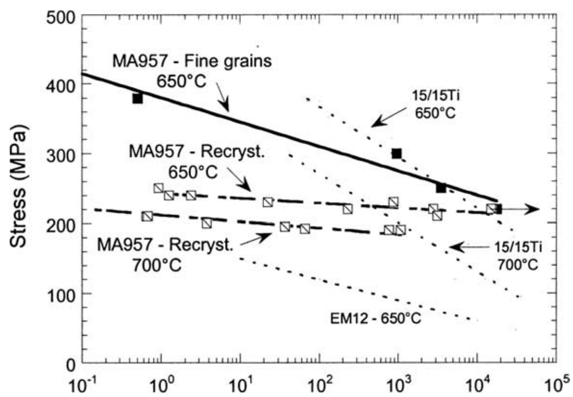

Fig. 4. Creep properties of MA957 ODS-alloy, obtained with both fine grain and recrystallized structures, and compared with the behaviour of the conventional Ti-stabilized austenitic stainless steel (15/15Ti) and the ferritic–martensitic 9Cr2MoVNb (EM12) steel.

stress σ/E, the values of σ_{th} were estimated at about 200 MPa at 650 °C and 170 MPa at 700 °C for a strain rate of 10^{-9}.

The physical origin of σ_{th} is not well known, but it may be related to the attractive interaction between dislocations and oxide particles [10–12].

7. Irradiation behaviour

To investigate the susceptibility to hardening and embrittlement induced by neutron irradiation, MA957 specimens were irradiated in the Osiris reactor at 325 °C for doses of 2 and 6 dpa.

Tensile properties were determined for samples irradiated in the as-extruded condition for both dose levels. Data obtained for MA957 alloy was compared with that established for the conventional martensitic steels irradiated in the same conditions, that is, MANET2 (10Cr0.6MoVNb) and F82H (7.5Cr2WTaV). Fig. 5 compares the irradiation-induced hardening, measured by the increase in yield stress of these materials, as a function of the dose. As expected, irradiation produced an increase of the tensile strength, but much lesser hardening is obtained for the ODS alloy (~200 MPa) than for the conventional steels. The initial yield strength of MA957 (818 MPa at 325 °C) is higher than that of F82H (456 MPa) and MANET2 (591 MPa) [3], but after 6 dpa MA957 presents a lower tensile strength than MANET2. Regarding ductility, a small decrease of elongation values are found for MA957, whereas a much greater decrease of these parameters is observed for the other materials as shown in Fig. 6. Moreover, values of reduction in area (ODS: 52%) are also higher after 6 dpa

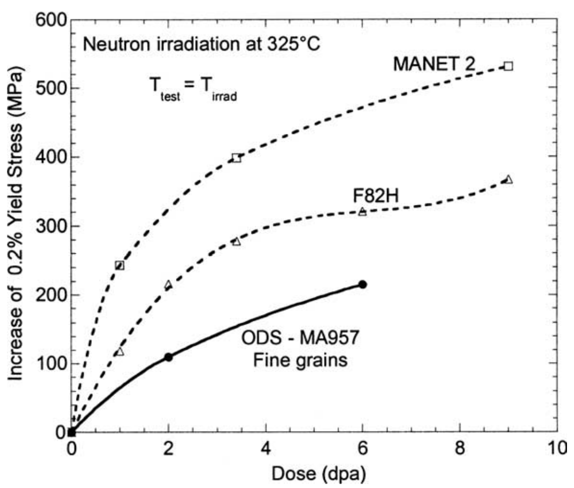

Fig. 5. Increase of yield stress as a function of dose for ODS-MA957 ferritic alloy, reduced activation F82H and conventional MANET 2 martensitic steels.

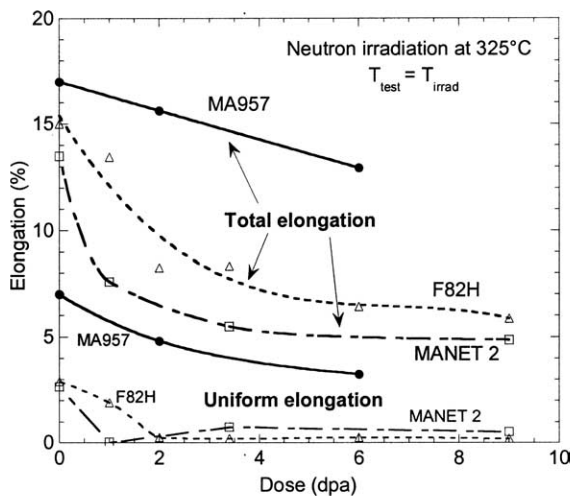

Fig. 6. Total and uniform elongation values as a function of dose for ODS-MA957 ferritic alloy, reduced activation F82H and conventional MANET 2 martensitic steels.

compared to the martensitic steels (F82H: 45%, MANET2: 30%). Thus, the irradiation behaviour of the ODS-MA957 is quite surprising considering its high Cr-concentration.

SANS techniques were used to study the changes in the nanostructure of the ODS-14%Cr/MA957 alloy induced by the neutron irradiation. Comparison of the scattered intensities for unirradiated and irradiated samples showed that no irradiation effects are observed in the oxide size distribution. Nevertheless, very fine precipitates, identified as the Cr-rich bcc α′-phase, are detected for 2 and 6 dpa. The mean radius of α′-particles slightly increases from 1.4 nm for 2 dpa up to 1.6 nm at 6 dpa, but their volume fraction of about 4.4% is not significantly changed with the increasing dose.

However, no simple correlation appears between the irradiation-induced hardening and the α′-precipitated fraction and/or radiation defects contributions. In the case of F82H and MANET2 steels, the large increase of the yield stress observed could not be attributed to α′-precipitation because no α′-phase was detected in F82H after a dose of 2.9 dpa and a smaller fraction is expected for MANET2 because of the lower Cr-content [4] compared to MA957. Further microstructural investigations are needed to evaluate each particular contribution to irradiation-induced hardening.

8. Conclusions

The mechanical behaviour of the ODS-14%Cr MA957 alloy was investigated for two different microstructures, that is, very fine grains and recrystallized intermediate grain structures.

Mechanical properties are very sensitive to microstructure. High tensile strength and very low DBTT values were found for the fine grain microstructure. But, this condition should have the disadvantage of some anisotropy of properties related to the anisotropic microstructure.

The recrystallized condition offers the potential of more isotropic behaviour. In particular, this condition presents higher ductility and better creep behaviour is expected for longer rupture times.

Very interesting results were obtained concerning the irradiation performances of the MA975 irradiated at 325 °C up to 6 dpa. Even the occurrence of a significant fraction of α′-phase (4.4%), this material exhibited lower hardening and excellent ductility after irradiation, compared to other structural material candidates such as reduced activation martensitic steel F82H.

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