Journal of Nuclear Materials 466 (2015) 286–291

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Hydrogen-induced crack nucleation in tensile testing of EUROFER 97 and ODS-EUROFER steels at elevated temperature

Evgenii Malitckii*, Yuriy Yagodzinskyy, Hannu Hänninen

Aalto University School of Engineering, Espoo, Finland

ARTICLE INFO

Article history:
Received 6 March 2015
Received in revised form
6 August 2015
Accepted 7 August 2015
Available online 12 August 2015

Keywords:
Hydrogen
ODS steel
Thermal desorption spectroscopy
Hydrogen-induced cracking

ABSTRACT

The effect of continuous hydrogen charging on tensile properties of EUROFER 97 and ODS-EUROFER steels was studied at room and elevated temperatures of 100 °C and 300 °C. The hydrogen effect decreases with increase of the temperature for ODS-EUROFER steel, while susceptibility to hydrogen of EUROFER 97 steel remains approximately the same at all testing temperatures.

Continuous hydrogen charging results in a reduction of the grain boundary cohesion of the EUROFER 97 and ODS-EUROFER steels tested at RT. With increase of the testing temperature up to 300 °C EUROFER 97 steel exhibits relatively high amount of micro-cracks which agglomerate in sub-micrometer size cracks, while the hydrogen-induced intergranular crack nucleation in ODS-EUROFER steel is effectively suppressed. Possible mechanism of the hydrogen-induced crack nucleation and propagation under applied external stress is discussed.

1. Introduction

The growing energy needs promote the development of new generation of the nuclear power plants. The enhanced safety, minimum radioactive waste generation and nuclear proliferation resistance are in the main focus as well as economic availability. In the most cases the adequate choice of structural materials for nuclear power plant units is based in their contribution for safety and durability. One of promising materials for application in Gen IV nuclear power plants and fusion reactors is reduced activation ferrite-martensite (RAFM) oxide dispersion-strengthened (ODS) steel because of its sufficient thermal creep strength, high tensile strength and resistivity to radiation-induced property degradation [1–3]. Friction stir welding is a possible method to fabricate joints of ODS-EUROFER steel avoiding the grain coarsening and agglomeration of yttria nano-particles that happens during standard fusion welding processes [4].

The large amount of dispersoid phase in ferrite-martensite matrix significantly increases the strength of the ODS alloys, but at the same time nano-sized oxide particles form approximately 0.3×10^5 (cm^2/cm^3) interface area that is considerable trapping site for hydrogen atoms as it was shown for ODS-EUROFER and PM2000 steels [5]. The high radiation doses in the active zones of the nuclear reactors result, inevitably, in hydrogen and/or helium accumulation due to the *(n, a)* and *(n, p)* transmutation reactions, and the accumulated atoms will be effectively trapped at the interfaces of the oxide nanoparticles. The trapped hydrogen significantly decreases the elongation to fracture of ODS-EUROFER steel resulting in intergranular fracture mode, while the trapped hydrogen does not affect strongly the mechanical properties of the base RAFM EUROFER 97 steel [6].

In order to simulate the real conditions of the operation the mechanical properties of EUROFER 97 and ODS-EUROFER steels were studied at temperatures from RT to 700 °C [2]. However, accumulated hydrogen and/or helium atoms may significantly change their thermal creep strength and ductility, namely reduction of elongation to fracture and tensile strength. Therefore, effect of continuous hydrogen charging on their mechanical properties has to be studied properly at elevated temperatures.

2. Experimental

The reduced activation ferrite-martensite EUROFER 97 and ODS-EUROFER steels were provided by Forschungszentrum Karlsruhe Institute for Materials Research [2] in the form of plates with size of about 6 × 65 × 65 mm. Table 1 shows the chemical composition of the studied steels.

Tensile specimens with gauge length of 4 × 2 × 0.2 mm were cut

* Corresponding author.
E-mail address: evgeny.malitskiy@aalto.fi (E. Malitckii).

http://dx.doi.org/10.1016/j.jnucmat.2015.08.016
0022-3115/

Table 1
Chemical composition of ODS-EUROFER and EUROFER 97 steels, wt.%.

	C	Si	Mn	Cr	Ni	Mo	Al	W	V	Ti	Co
ODS-EUROFER[a]	0.086	0.03	0.39	9.2	0.02	0.0056	0.003	1.14	0.1965	<0.003	0.0036
EUROFER 97	0.11	0.03	0.55	8.95	0.013	<0.005	0.009	1.06	0.202	<0.003	0.004

[a] Mechanically alloyed with addition of 0.3 wt.% yttrium oxide (Y_2O_3).

from the plates transverse to the rolling direction. The specimens were mechanically polished finishing with silicon carbide paper #2000. Shape of the tensile specimens is shown in insert of Fig. 2.

Thermal desorption spectroscopy (TDS) of hydrogen release was performed after hydrogen charging from a glow discharge plasma at potential of 30 V using the hydrogen charging process instrument. The hydrogen ion flux was measured to be 1.6×10^{16} $cm^{-2}s^{-1}$ [7]. The time of plasma exposure is about 2 h. The temperature range of TDS is from RT to 1000 K and heating rate is about 6 K/min.

The tensile testing of the studied steels was carried out in both as-supplied and continuous hydrogen charging (denoted as H-charging) conditions and at temperatures of RT, 100 °C and 300 °C. Plasma charging apparatus was combined with 2 kN DEBEN tensile test machine, and hydrogen charging was performed continuously during the tensile test at the respective temperatures after 2 h of hydrogen pre-charging. The strain rate of tensile testing is about 10^{-4} s^{-1}.

The side surfaces of the tensile specimens tested in as-supplied condition and under continuous H-charging were polished finishing with Hitachi IM4000 ion milling system. The ion milling system allows to minimize the resultant deformation layer of the grinding revealing the damage of the interior of the tensile test specimens. The microstructures of the interior close to the fracture surface of the tensile test specimens were studied by FEG-SEM Zeiss 55 Ultra.

3. Results and discussion

In the earlier investigations of ferrite-martensite EUROFER 97 and ODS-EUROFER steels it has been shown that the grain structure with mean grain size of about 1 μm is typical for both materials [5]. The chromium and tungsten carbides with mean size of about 100 nm were preferably observed at the grain boundaries of the studied steels. The mechanical alloying of the ferrite-martensite steel with addition of Y_2O_3 nano-sized particles results in their homogeneous distribution within the steel grains with a mean density of about 1×10^{22} m^{-3}. The mean size of the dispersoid phase particles was about 25 nm [5]. The non-metallic inclusions (denoted as NMIs) of Al, Ti and V oxides were observed in ODS-EUROFER steel in a higher amount than in EUROFER 97 steel resulting, probably, from the oxygen from yttrium oxides during the steel processing [6].

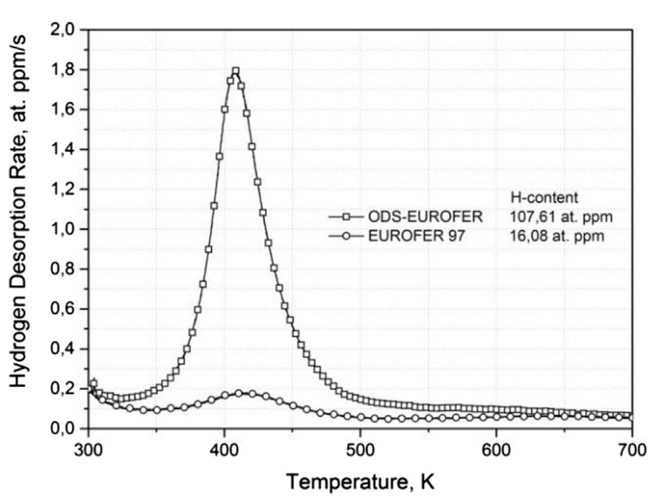

Fig. 1. TDS curves of hydrogen release from EUROFER 97 and ODS-EUROFER steels after H-charging at 50 °C for 2 h.

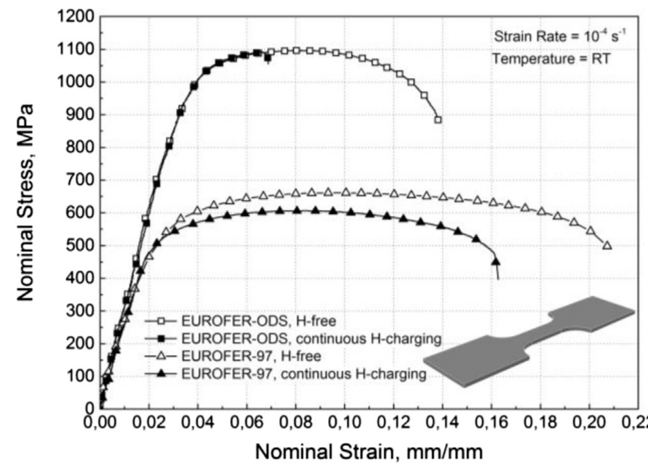

Fig. 2. CERT stress–strain curves of ODS-EUROFER and EUROFER 97 steels obtained at RT. Shape of the tensile specimen is shown in the insert.

Results of hydrogen release and detrapping of the studied steels are shown in Fig. 1. ODS-EUROFEER and EUROFER 97 steels manifest rather different ability to hydrogen uptake after 2 h of the hydrogen plasma charging at temperature of 50 °C. Total hydrogen concentration of ODS-EUROFER steel was about 107.6 at. ppm that was significantly higher than 16.1 at. ppm of hydrogen of EUROFER 97 steel. In spite of different ability to hydrogen uptake and trapping, both studied steels seem sensitive to a continuous hydrogen charging at RT. Typical constant extension (CERT) stress–strain curves (see Fig. 2) of both continuously H-charged steels show a significant reduction of elongation to fracture, while only EUROFER 97 steel suffers a slight reduction of the tensile strength.

The influence of continuous H-charging on tensile properties of ODS-EUROFER and EUROFER 97 steels was studied at elevated temperatures. Both steels were tested in both H-free and continuous H-charging conditions at temperatures of about 100 and 300 °C. The results are summarized in Fig. 3 and compared with those obtained at RT. The elongation to fracture of the studied steels tends to be less sensitive to continuous hydrogen charging at temperature of 300 °C than at RT. However, the slight hydrogen effect on tensile strength of both studied materials looks independent of temperature. The parameter of sensitivity to hydrogen (Eq. (1)) is plotted against the testing temperature in Fig. 3:

$$\delta_H = (\varepsilon - \varepsilon_H)/\varepsilon \quad (1)$$

where ε – total elongation to fracture of H-free specimen and ε_H – total elongation to fracture of continuously H-charged specimen.

Fig. 3 indicates that the hydrogen effect on the mechanical properties of ODS-EUROFER steel decreases with increase of the