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Hydrogen embrittlement and fracture mode of EUROFER 97 ferritic-martensitic steel

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ABSTRACT

The hydrogen embrittlement of EUROFER 97 ferritic-martensitic steel promotes a change from ductile to brittle trans- and inter-granular fracture during fracture toughness testing. The brittle fracture is controlled by hydrogen content in the range of 2–4 wppm and strongly depends on the extent of hydrogen saturated trapping sites. The same hydrogen concentration in base and weld metals manifests itself by different extents of brittle fracture, higher in base metal and lower in weld metal as a result of a different number and saturation of trapping sites. The extent of brittle fracture on surface specimens decreases along the hydrogen concentration gradient from the crack tip and with increasing testing temperature.

1. Introduction

Reduced activation ferritic-martensitic steels EUROFER 97 will be used for structural components of the Test Blanket Module (TBM) in the ITER fusion reactor. In plasma facing materials exposure to higher energy neutrons results in transmutation reactions generating hydrogen and helium. The main source of hydrogen in structural materials is a transmutation reaction, e.g. (n,p) and hydrogen ingress into material from external sources like hydrogen isotope implementation from the plasma side as well as tritium permeation from breeder material into the TBM structure [1,2]. The increase of hydrogen content can influence the degradation of mechanical and fracture properties and leads to hydrogen embrittlement. The susceptibility of ferritic-martensitic steels was investigated by means of tensile and constant extension rate tests and low cycle fatigue tests. Only a limited number of data of hydrogen effect on a fracture toughness are available. In our paper the effects of hydrogen on static fracture toughness of EUROFER 97 steel have been measured and evaluated. It was observed that increased hydrogen content resulted in the change of failure mechanism during the transition from ductile to brittle fracture. Therefore the goal of the present work has been the assessment of fractographic observations and evaluations of failure mechanisms of the ferritic-martensitic steel charged on a different level of hydrogen content.

2. Effect of hydrogen in steel

One of the main problems of using ferritic-martensitic steels as structural materials of TBM is hydrogen embrittlement affected by hydrogen uptake, leading to subsequent degradation of mechanical and fracture properties [3,4]. The hydrogen embrittlement is largely affected by temperatures and can be substantially lower above 300 °C as a result of increased diffusivity and lower number of trapping sites. At lower temperatures approximately below 200 °C, the hydrogen content and diffusivity are significantly dependent on the presence of trapping sites, such as inclusions, precipitates, grain boundaries, lath boundaries, dislocations, vacancies, and microcracks. The trapping sites act as reversible or irreversible traps, i.e. could release or store hydrogen. The reversible traps efficiency decreases along with the temperature of material annealing depending on the recovery process. There is a considerable uptake of hydrogen due to irradiation-induced traps that, according to some evaluations, can reach several dozens of wppm in the Water Cooled Lead Lithium Blanket [5,6].

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The effect of hydrogen on structural materials is usually investigated depending upon its content after electrolytic charging in H_2SO_4 solution or charging in hydrogen atmosphere. The experiments with ferritic-martensitic steels show that hydrogen charging could take place both under low current density lasting even several days (4 mA/cm^2 for 72 h, Maday [7]) and higher current densities up to 10 mA/cm^2 for a shorter period of time [8]. These methods enabled hydrogen content in the sample to reach up to ~8 wppm.

Ferritic-martensitic steels as TBM potential materials have been investigated regarding the effect of increased hydrogen content. Hydrogen embrittlement of EUROFER 97 manifests itself at dynamic charging and room temperature (RT) already with 1–2 wppm; Maday [7] gives 1.6 wppm. In her work, EUROFER 97 and T 91 (9CrMoVNb) steel investigated at slow strain rate test exhibit decreased reduction of area at 1–2 wppm and reveal similar intergranular fracture. In the case of some ferritic-martensitic steels, such as F82H steel and Manet II tested at low strain rate, embrittlement takes place with 1–2 and 3–4 wppm [2]. The ferritic-martensitic F82H steel was also investigated in terms of the effect of loading mode and hydrogen content of 4 ppm on the fracture toughness. The presence of hydrogen has decreased both fracture toughness (J-integral) and tearing modulus. The minimum mixed-mode fracture toughness with presence of hydrogen was about 30% of the value of the uncharged steel [9].

3. Material and methods

The investigations were performed with EUROFER 97 steel manufactured for EFDA (European Fusion Development Agreement) association Steel plates of 300 × 300 × 14 mm from the EUROFER segment 4/15, heat No. E 83698 were delivered. A weld joint was prepared from two plates 14 × 90 × 300 mm by the TIG method with EUROFER filler wire of Ø 1 mm in the CEA Saclay [10]. Material chemical composition is (by w%): C 0.11, Cr 8.82, V 0.20, W 1.09, Ta 0.13, Ni 0.022, Mn 0.47, Si 0.04, P 0.005, S 0.004. Microstructures of base metal and weld metal are given in Fig. 1.

The testing was performed with micro-TPB (three-point bend) specimens of 3 × 4 × 27 mm machined from plates and weld joint with notch orientation parallel to the rolling direction and axial axis in transverse direction. Specimens were side grooved and precracked before hydrogen charging. The notches and side grooves were machined using a 45°saw of cutting edge diameter 0.3 mm. MicroTPB specimens were cathodic charged for 2 h in 0.5 M H_2SO_4 solution with the addition of 0.5 g/l $NaAsO_2$ and with current density in the range of 10–150 mA/cm^2 at the temperature of 75 °C. After charging the specimens were stored in liquid nitrogen until the tests of fracture toughness were started. After testing and specimens final fracture, one half of the specimens was kept in liquid nitrogen before hydrogen analysis, the second half was used for specimen fracture surface observation.

The three-point bend method, the compliance method as well as the hydraulic tensile testing machine were used for measurements. The testing and results evaluation were carried out in agreement with ASTM E 1820-01 [11] and ESIS standard [12]. The critical value of *J*-integral $J_{0.2}$ has been evaluated. J-integral experimental points and crack growth values of JR curve were adjusted by a power regression curve and $J_{0.2}$ values were measured at the point of intersection of J–R curve and 0.2 mm exclusion line.

4. Results

Ferritic-martensitic steel EUROFER 97 in the as-received condition contains 0.3–0.4 wppm of hydrogen. The conditions of hydrogen trapping in specimens during the tests were evaluated by the measurement of hydrogen content after electrolytic charging and after releasing from specimens at temperatures of 20–120 and 150 °C. The results given in [13] show that the hydrogen contents after charging attained were two times higher in weld metal in comparison with base metal. A considerable release below 2 wppm was obvious for about 1 h at higher temperatures.

Fracture surfaces of specimens were characterized by the pre-crack, crack growth and final fracture areas. The pre-crack and final fracture areas did not exhibit any changes of observed specimens. Crack growth fractographic assessments were therefore performed on the area of crack initiation, the central area of crack growth and the area of near final fracture (Table 1). Ductile dimple failure was only observed in base and weld metal specimens tested in the as-received state as shown for base metal in (Fig. 2).

4.1. Testing at room temperature

For base metal, hydrogen induced loss of ductility was observed for specimens containing 2.0 and 4 wppm of hydrogen (Figs. 3 and 4). The area of crack initiation showed only a brittle trans-granular (TG) and inter-granular (IG) fracture for both hydrogen contents. In the central area, the failure mechanism at lower hydrogen content of 2 wppm manifested itself by both inter-granular and ductile dimple fracture in the same extent, while at higher 4 wppm content only inter-granular and brittle trans-granular fractures were observed.

In the near final fracture area at 2 wppm only ductile dimple fracture was observed At 4 wppm the mixed mode of brittle fracture and ductile behaviour were observed while brittle fractures prevailed over ductile dimple failure.

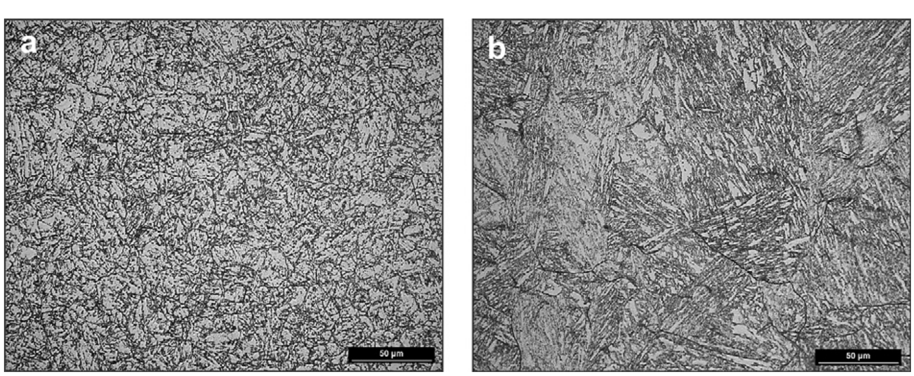

Fig. 1. Microstructure of (a) base metal and (b) weld metal.