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Experimental investigation on deuterium effects on the tensile performance of two types of China Reduced Activation Ferritic-Martensitic steels

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ABSTRACT

The present work is aimed to study on the effect of deuterium on mechanical properties of two martensitic steel, i.e. CLAM steel and CLF-1 steel. Tensile tests were operated at room temperature before and after deuterium charging. The results showed the ultimate tensile stress and area reduction coefficient of both steels were decreased after deuterium charging. The shrinkage rates of hydrogen induced reduction of area of CLAM steel and CLF-1 steel were 18.65% and 45.98%, respectively, which showed poor resistance to hydrogen embrittlement. This may be related to a lot of lath martensite phase in both steels. And the mechanism of even more sensitive to hydrogen damage for CLF-1 steel is probably due to its finer prior austenite and lath martensite phase, higher contents of carbide particles, as well as more hydrogen content and lower diffusivity.

1. Introduction

Reduced Activation Ferritic/Martensitic (RAFM) steels have been proposed as the most promising candidate structural materials for the experimental fusion reactor ITER, instead of austenitic steels owing to their better swelling resistance, improved irradiation resistance and favorable thermo-physical properties [1]. However, during ITER normal operation, the structural materials will be irradiated by various particles, particularly deuterium (D) and tritium (T) can be preliminary concluded that their contents might reach rather high values due to high hydrogen diffusivity and trapping site concentration [2]. Moreover, additional external hydrogen sources (e.g. hydrogen produced by (n, p) transmutation reaction, aqueous corrosion, hydrogen added to cooling or purging gas, radiolysis of cooling water) can't be ignored [3]. As a result of hydrogen accumulation, a degradation of mechanical performance like the structure strength and ductility would occur for the metal. Therefore, hydrogen embrittlement (HE) susceptibility must be investigated for the assessment of service lifetime of fusion reactor.

In the past few decades, research on RAFM steels has been investigated all over the world by means of tensile and impact tests, as well as low cycle fatigue tests [4–6]. However, only a limited number of data of deuterium on a fracture toughness, especially for two types of China Reduced Activation Ferritic-Martensitic steels, namely CLAM steel [7] and CLF-1 steel [8] are available. In the present paper, the effects of deuterium on the tensile performance of CLAM steel and CLF-1 steel have been measured and evaluated. The study was based on quasi-tensile tests conducted at room temperature using standard cylinder samples. HE susceptibility was investigated on the basis of the ultimate tensile stress, the area reduction coefficient and fracture morphology.

2. Experimental

2.1. Sample preparation

The materials used were CLAM steel supplied by the Institute of Nuclear Energy Safety Technology and CLF-1 steel obtained from Southwestern Institute of Physics. Their measured chemical compositions determined by glow discharge spectrometry were listed in Table 1, and they are basically in coincidence with the previous research [9,10]. In addition, the as-received state heat treatment conditions for both steels were shown in Table 2. All specimens were fabricated into regular shapes with 10 mm × 10 mm × 2 mm for microstructure observation, while the tensile specimens were designed according to China national standard GB/T228.1–2010, which was shown in Fig. 1.

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Table 1
Main elemental composition of CLAM and CLF-1 in wt. %.

Element	Fe	Cr	W	V	C	Mn	Ta	O	Si
CLAM	Balance	8.87	1.48	0.21	0.09	0.47	0.10	–	0.05
CLF-1	Balance	8.41	1.51	0.25	0.12	0.47	0.12	0.001	–

Table 2
Heat treatment conditions for CLAM and CLF-1 steels.

Type of steel	Normalization	Tempering
CLAM	1253 K /30 min/ air cool	1033 K/90 min/ air cool
CLF-1	1253 K /45 min/air cool	1013 K/90 min/ air cool

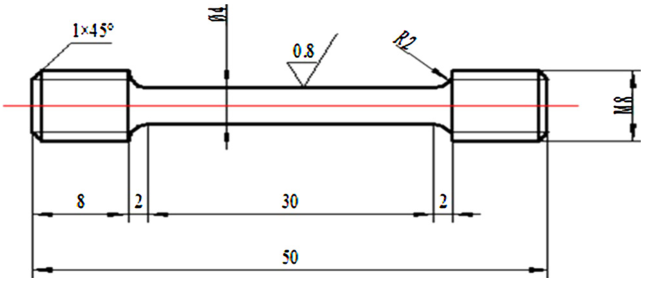

Fig. 1. Shape and dimensions of a smooth bar tensile specimen.

2.2. *Materials characterization*

To reveal the undeformed microstructure and carbide distribution for the optical microscopy (OM) and SEM studies, the surface of the samples after mechanical polishing was etched by Villella's reagent (1 g of picric acid, 5 ml of hydrochloric acid and 100 ml of ethyl alcohol). The field emission high resolution transmission electron microscope (F20) was applied to better observe the internal microstructure of CLAM and CLF-1 steels. TEM samples were electrolytically thinned using a solution of 5% $HClO_4$-95% C_2H_5OH. For determining whether there was remaining austenite, the X-ray diffraction (XRD) examination for CLAM and CLF-1 steels was carried out. And the software (Image Pro Plus 6.0) was applied to aid the statistical analysis of the precipitate size and number of both steels.

2.3. *Tensile test*

Three types of specimens for tensile test were prepared in this study. The first type, denoted as the [deuterium-charging] specimen, was exposed to D_2 with a pressure of 5×10^5 Pa at 773 K for 10 h to obtain D saturated RAFM steels according to the diffusive transport parameters of D_2 through both steels [2], and the schematic diagram of gas exposure was shown in Ref. [11]. Prior to deuterium charging, the samples surface were polished with #800 emery paper to remove the oxide layer. The deuterium source used was high purity of 99.999%. Immediately after deuterium charging, all samples were kept in D_2 gas and water-cooling to R.T. The second type, denoted as [non-charged + 773 K] specimen without deuterium per-charging, was annealed at 773 K for 10 h in a vacuum of 10^{-5} Pa to match the thermal history. The third type was as-received samples without any treatment in order to characterize the tensile behavior of the basic material.

The tensile tests were performed on the universal testing machine (CMT5105). Previous studies [12,13] have reported hydrogen had less effect on HE susceptibility at strain rates upon the order of 10^{-3} s^{-1}. In contrast, hydrogen decreased the relative reduction of area at strain rates below the order of 10^{-4} s^{-1}. Therefore in this study, the initial strain rate was 2.78×10^{-4} s^{-1} for [deuterium-charging] specimen while 5.56×10^{-4} s^{-1} for [non-charged + 773 K] specimen and as-received specimen. Each test was repeated three times respectively to improve the reliability of the experimental data, subsequently the average was compared. And only one test was shown not to overload the graph. All the tensile tests were performed at room temperature. The SEM was employed to characterize the fracture morphologies.

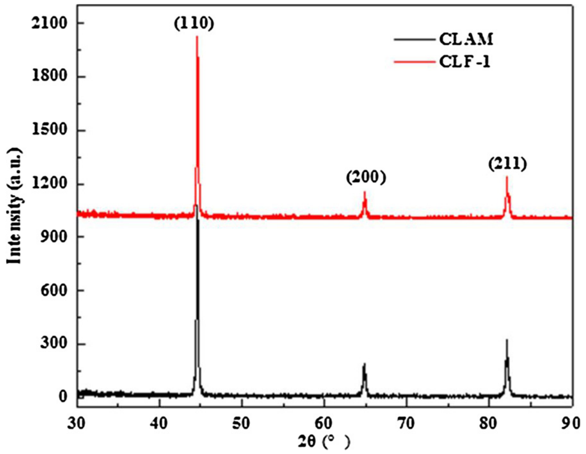

Fig. 2. XRD diagram for CLAM steel and CLF-1 steel.

After tensile tests, fracture section diameter was carefully measured to evaluate the area reduction coefficient *Z%*, which was considered to be more sensitive than the coefficient of elongation *A%* [14,15]. In this paper, the shrinkage rate of hydrogen induced reduction of area Z_L was taken as the hydrogen embrittlement sensitivity index as follows:

$$Z_L = \frac{Z_0 - Z_H}{Z_0} \times 100\%$$

Where Z_0 is the reduction of area before deuterium charging, and Z_H represents the reduction of area after deuterium charging.

3. Results and discussion

3.1. *Characterization of the undeformed microstructure*

Fig. 2 shows the XRD diagrams for CLAM and CLF-1 steels, respectively. Both steels have only a single phase, which is identified as full martensite phase transformed from austenite during quenching. The microstructure of both steels in the as-received state are presented in Fig. 3. For CLAM steel, the equiaxed prior austenitic grain size is in the range of 10–20 μm, while 40–60 μm for CLF-1 steel. Furthermore, a significantly larger number of precipitates are observed in CLF-1 steel compared with CLAM steel, which has been also confirmed by SEM observations of surfaces as shown in Figs. 4 and 5. It is noted that only one SEM image for each steel (Fig. 4) is shown not to overload the graph, despite three different images are used for each steel to analysis carbide distribution and the sum (Fig. 5) is compared. In these SEM images, the precipitates exhibit a strong bright contrast. The authors suspected that shorter normalization time inhibits the austenite grain growth and effective diffusion of carbide-forming elements (Cr, W and V) for CLAM steel [4,16]. Remarkedly, there is no peak of the precipitates in the XRD profile, as a result of less content of carbides. However, carbides of CLF-1 steel are extracted electrolytically and their X-ray diffraction patterns are obtained [10].

The bright-field (BF) TEM images in Fig. 6 show lath structure in both steels, and the average lath width of CLF-1 steel is 0.4 μm, while 0.5 μm for CLAM steel. Additionaly, carbides are distributed along the lath interface and grain boundary of both steels and the electronic diffraction pattern shows that these precipitates are mainly $M_{23}C_6$. The chemical compositions of the martensite and carbides are presented in Fig. 6(e) and (f), respectively. It shows that the carbides are Cr-rich phase which might be $(Fe, Cr, W)_{23}C_6$ [17].