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Deuterium retention removal in China reduced activation ferritic-martensitic steels through thermal desorption and hydrogen isotope exchange

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ABSTRACT

Effective removal of deuterium and tritium trapping in fusion reactor materials is of great importance both to recycle fuel and prevent the performance degradation. Experiments on deuterium removal in China Low Activation Martensitic (CLAM) steel and China Low-activation Ferritic (CLF-1) steel through thermal desorption and hydrogen isotope exchange have been performed. The results indicate that CLAM steel has only one desorption peak at 578 K with an activation energy of 21 kJ/mol, while CLF-1 steel has two desorption peaks, whose corresponding activation energy is 31 kJ/mol at 563 K and 94 kJ/mol at 797 K respectively. After 2 h thermal desorption, deuterium removal efficiency for CLAM is more than 90% above 373 K. Yet for CLF-1, deuterium removal efficiency is less than 90% below 573 K. Hydrogen isotope exchange can slightly enhance deuterium release. Overall, Deuterium retention in both materials can be removed effectively at relatively low temperature (< 673 K) through thermal desorption due to the high hydrogen diffusivity, especially for CLAM steels.

1. Introduction

In the long pulse magnetic confinement fusion devices such as ITER and DEMO, permeation and retention behavior of hydrogen isotopes, deuterium (D) and tritium (T) in Reduced Activation.

Ferritic-Martensitic (RAFM) steels are vital for the economy and safety of fusion reactors [1–5]. Except for T supplied from outside, T can be generated by energetic neutrons and subsequent (n, p) or (n, α) transmutation of some non-metallic impurities like protium (H) or nitrogen (N). Moreover, T escapes from the plasma region and enters the structural material, producing He-3, which can destroy the performance of the structural materials, such as thermal conductivity, swelling and so on. It is difficult to deal with a lot of D/T during the decommissioning of fusion devices. Therefore, it is necessary to develop an effective technology for D/T removal. But T_2 is difficult to obtain as a strategic resource, thus it is replaced by D_2 in this study due to their similar properties. In the present studies, thermal desorption is mainly used for the treatment of D, which makes D atoms retention in materials gather again by rising the temperature, subsequently release to achieve the purpose of decontamination. While the hydrogen isotope exchange method is also put forward by some researchers [6,7], which makes the hydrogen atoms or molecules occupy the D capture defect in the materials, so as to enhance decontamination effect.

As the most promising candidate structure materials of fusion reactor, RAFM steels are researched in various countries [8–12]. China has developed its own RAFMs, namely, China Low Activation Martensitic (CLAM) steel [13] and China Low-activation Ferritic (CLF-1) steel [14]. In this paper, two different ways, thermal desorption and hydrogen isotope exchange are respectively used to remove D from both steels, and D removal efficiency is compared.

2. Experimental

2.1. Sample preparation

CLAM steel was supplied by the Institute of Nuclear Energy Safety Technology and CLF-1 steel obtained from Southwestern Institute of Physics. The main elemental compositions are shown in Table 1, as well as detailed heat treatment conditions for both steels in Table 2. In order to improve comparability and minimize the influence of micro-structural effects, all samples were fabricated into disks with Φ 20 mm × 2 mm thick from the same manufacturing batch. The surfaces were mechanically polished to a mirror-like finish then ultrasonically cleaned in ethanol and acetone. Annealing was subsequently carried

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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	N	
CLAM	Balance	9.0	1.5	0.2	0.1	0.45	0.07	–	0.01	0.02	[13]
CLF-1	Balance	8.5	1.6	0.3	0.12	0.6	0.1	0.001	–	–	[14]

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

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

out at 773 K for 5 h in a vacuum of 10^{-5} Pa to relieve residual stresses.

2.2. Methods

The microstructures of both steels were investigated by optical microscopy (OM) and scanning electron microscopy (SEM: Sirion 200). The samples for average size estimation were etched using a solution of $C_6H_3N_3O_7$-HCl-C_2H_5OH.

The schematic diagram of gas exposure was shown in Fig. 1. D_2 was stored in Zr-Co storage bed, which was heated to drive D_2 into the standard vessel. The pipe system was cleaned three times by 10^4 Pa D_2 before each experiment. The samples were exposed to D_2 with a pressure of 5×10^5 Pa at 773 K for 5 h to obtain D saturated RAFM steels according to the diffusive transport parameters of D_2 through both steels [15]. After exposure, thermal desorption or hydrogen isotope exchange was carried out. In the process of thermal desorption, the D saturated samples were respectively kept for 2 h in a vacuum of 5 Pa at 373 K, 473 K, 573 K and 673 K, while for hydrogen isotope exchange, they were respectively kept for 2 h in H_2 atmosphere at 10^5 Pa and 373 K, 473 K, 573 K and 673 K.

After D removal, all samples were cooled rapidly to R.T, Subsequently TDS experiments were followed. The 316 L stainless steel pipe which ions passed through was baked for 72 h to remove residual gas in the materials before TDS experiment. The samples were heated linearly up to 1273 K at 10 K/min in the quartz tube, with a vacuum of 10^{-5} Pa. In addition, in order to calculate the activation energy of every D_2 desorption peak, the heating rate was 5 K/min, 10 K/min, 15 K/min and 20 K/min respectively for the D saturated RAFM steels without D removal. D_2 ($m/z = 4$) and HD ($m/z = 3$) signal was tracked by quadrupole mass spectrometer (QMS). And the D_2/H_2 signal was calibrated using two standard leaks after experiment. Thus, HD signal could be theoretically calibrated, which was the average of the sum of D_2 and H_2.

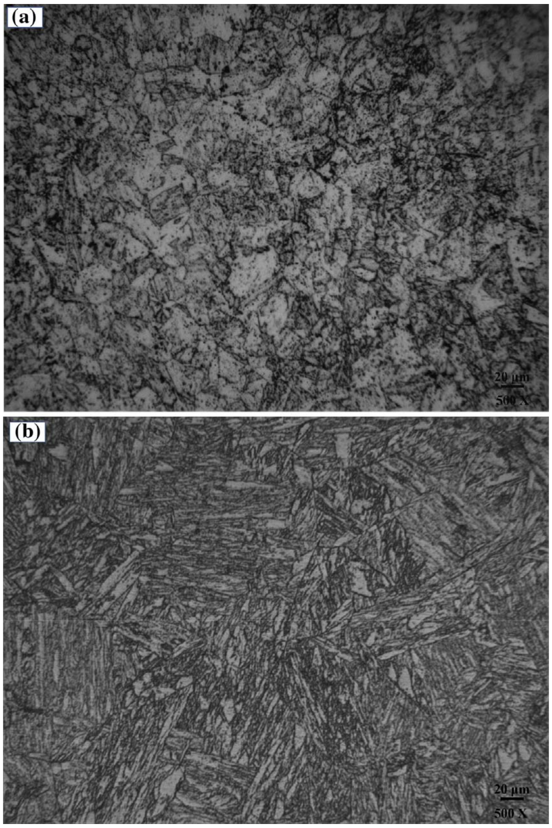

Fig. 2. Comparison of microstructure between (a) CLAM steel and (b) CLF-1 steel.

3. Results and discussion

The microstructure of CLAM steel and CLF-1 steel after heat

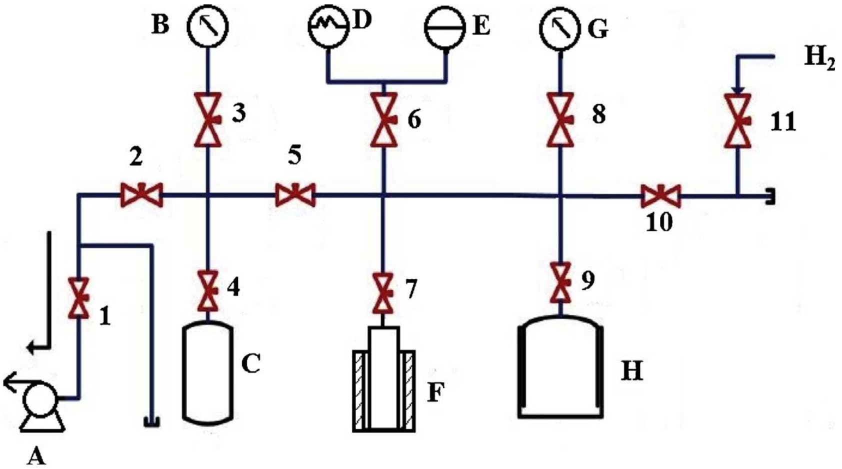

Fig. 1. Schematic diagram of gas exposure. A: scroll pump, B: pressure gauge (~1 bar), C: standard vessel, D: resistive vacuum gauge, E: film vacuum gauge, F: apparatus, G: pressure gauge (~10 bar), H: Zr-Co alloy storage bed, 1–11: Swagelok valves.