



Tritium permeation characterization of Al₂O₃/FeAl coatings as tritium permeation barriers on 321 type stainless steel containers



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HIGHLIGHTS

- T-permeation has been measured on bare and coated type 321 SS containers.
- Al₂O₃/FeAl coating give a reduction of T-permeability of 3 orders of magnitude.
- Mechanism of Al₂O₃/FeAl barrier resisting T-permeation has obtained.
- Quality of TPB is a very important factor for efficient T-permeating reduction.

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ABSTRACT

Accurate tritium transport properties of prospective tritium permeation barriers (TPBs) are essential to tritium systems in fusion reactors. By passing a temperature and rate-controlled sweeping gas over specimen surfaces to carry the permeated tritium to an ion chamber, the gas-driven permeation of tritium has been performed on 321 type stainless steel containers with Al₂O₃/FeAl barriers, to determine the T-permeation resistant performance and mechanism of the barrier. The tritium permeability of the Al₂O₃/FeAl coated container was reduced by 3 orders of magnitude at 500–700 °C by contrast with that of the bare one, which meets the requirement of the tritium permeation reduction factor (PRF) of TPBs for tritium operating components in the CN-HCCB TBM. The Al₂O₃/FeAl barrier resists the tritium permeation by the diffusion in the bulk substrate at a limited number of defect sites with an effective area and thickness, suggesting that the TPB quality is a very important factor for efficient T-permeation resistance.

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1. Introduction

In future fusion reactors, the tritium handling amount will be several kilograms per hour between the plasma burning vessel and the fuel cycling system, beyond of the tritium handling capacities of any currently existing tritium facilities in the world. One of the technical challenges to safely process such a huge tritium amount is the efficient tritium confinement within the vessels and pipes, due to the high leakage and permeation of tritium [1]. At a typical temperature of 500 °C, the tritium loss by permeation through containers is far more significant than that by a natural leakage from joint points, because structural materials, such as Eurofer97, CLAM and F82H ferritic-martensitic steels, 316L and 321 austenite

stainless steels, having high permeabilities of hydrogen isotopes [2]. Moreover, the mechanical performance of structural materials will be degraded, due to the hydrogen embrittlement and tritium decayed helium damage. Thus suppression of tritium permeation through and into structural material of ducts and components is one of key issues for tritium self-sufficiency and safety for fusion reactors [2,3].

Externally applied materials, having lower tritium permeabilities than the substrates, on structural materials have been examined as tritium permeation barriers (TPBs). Leading TPBs, such as aluminum rich coatings, TiN/TiC coatings and SiC coatings, etc, have been shown reductions of hydrogen isotopic permeabilities by factors of 10 to over 10⁴ [4]. Up to present, aluminum rich coatings, consisting of aluminide layers with Al₂O₃ scales, have been selected as one of the prior developed TPBs for TBMs by Europe, China and India for their high permeation reduction effectiveness, low thermal mismatch, excellent compatibility and self-healing [5–10]. Such aluminide coatings with Al₂O₃ scale can be fabricated by

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aluminization of steels with hot-dip aluminizing (HDA), pack cementation (PC), vacuum plasma spray (VPS) or electrochemical deposition (ECD), followed by aluminization and oxidation. The inner aluminide layer consists of mixed Fe_3Al and FeAl . Ni, Cr, and mixed-aluminides also forms. The inner layer often has a mixed oxide scalarich in Al_2O_3 . Coatings produced by HDA followed by heat treatment on DIN 1.4914 and EUROFER 97 steels [5–7] at KIT, Germany, by PC and chemical vapor deposition (CVD) on EUROFER 97 steels [5,11] at CEA, France and by VPS on MANET steels [5] at JRC, Italy reduced hydrogen or deuterium permeability by a factor of 1500–3100, 1300–3100 and 400–1000, respectively in a temperature range from 300 to 450 °C. HDA and PC were applied to tubes ($\Phi 29 \times 1.5$ mm, 100 mm long), reducing the hydrogen permeability by a factor of 15–140 and 6–15, which partly qualified for the gas-phase [5,12]. $\text{FeAl}/\text{Al}_2\text{O}_3$ coatings on a 316L tubular specimen ($\Phi 10 \times 3.0$ mm, 1000 mm long) by PC followed by oxidation in CIAE, China achieved a deuterium PRF of 3000 in the gas-phase at 550 °C [13]. We prepared a high quality $\text{FeAl}/\text{Al}_2\text{O}_3$ coating, with a 3-step process of ECA followed by aluminization and selective oxidation, on 321 type stainless steel containers ($\Phi 80 \times 2.0$ mm, 150 mm long), and the obtained deuterium PRF varied from 113 to 2262 at 600–727 °C [9,14,15]. To describe the hydrogen isotope interactions with TPBs, composite diffusion models, i.e., the area defect model and surface desorption model were proposed [2,4], suggesting that the hydrogen permeation was controlled by the hydrogen molecule dissociation rate on the barrier surface or by the hydrogen atom diffusion in the barrier. It was found, from above literatures, that tritium permeation characterizations were evaluated from H/D-permeation tests, and PRFs for hydrogen and deuterium were reported to be many thousands in certain barrier systems, which did guide the process specification for the quality of TPBs.

However, with currently focusing on the next milestones of the conceptual design of TBM systems, in view of practical fusion applications, the safety demonstration urgently requires work on the interpolation and safety assessment of pre-designed systems, which in turn requires new experimental data [16]. “Real” tritium transport properties of TPBs in specific conditions of tritium components are critical parameters for the consolidating design and safety assessment of tritium systems. Current H (D) properties typically could not provide accurate predictions for tritium, since it is a radioactive hydrogen isotope with low energy beta radiation. In particular, few information can be found in literatures devoting tritium transport properties for aluminum rich TPB coatings [17]. Therefore, there will be considerable uncertainties for tritium transport properties of TPBs, since the current pre-analysis of the design and safety assessment of tritium systems is almost based on the properties of H or D. Moreover, the data available in literatures are quite limited. There is a significant evidence that the effectiveness of TPBs decreased in radiation environments performed in the HFR Petten reactor in the Netherlands [18,19], which raised considerable concerns for the effective performance of TPBs under beta radiation, followed by continuous tritium disintegration into ^3He . Thus, the design of efficient T-permeation resistive components, with extended lifecycles, for tritium systems urgently relies on accurate tritium transport properties of freshly prepared and tritium aged TPBs. This is important to ensure the tritium release value into the environment of CN-HCCB below 0.1Ci/FPd [9].

In the present work, gas-driven permeations of tritium have been carried out on $\text{Al}_2\text{O}_3/\text{FeAl}$ coated 321 type stainless steel containers, with the permeation temperatures varying from 500 to 700 °C and tritium pressures from 30 to 40 kPa, to determine the tritium permeation resistance and corresponding mechanism of $\text{Al}_2\text{O}_3/\text{FeAl}$ barriers.

2. Experimental details

2.1. Specimen preparation

Bare and $\text{Al}_2\text{O}_3/\text{FeAl}$ coated 321 type austenitic stainless steel containers, with a nominal composition of Fe–18Cr–9Ni–0.46Ti–0.05C (mass fraction, %), were used as test specimens. The containers were one end closed tubes, with a length of about 150 mm, an external diameter of 80 mm, and a thickness of 2.0 mm.

The $\text{Al}_2\text{O}_3/\text{FeAl}$ coating, consisted of an outer $\gamma\text{-Al}_2\text{O}_3$ layer with a thickness of 0.2 μm and an inner (Fe,Cr,Ni)Al/(Fe,Cr,Ni)₃Al layer of 50 μm thickness, on the outer surface of the container specimens was fabricated by a 3-step process of ECD followed by aluminizing and selective oxidation as reported previously [14,15], which was briefly described as follows. Firstly, the container specimen was mechanically ground using SiC polishing agent down to 0.5 μm , and then electrochemically etched by anodic polarization to remove the surface oxidation layer and obtain a smooth and reproducible surface for Al deposition. The Al deposition from a Lewis acidic mixture (molar ratio 1:2) of 1-ethyl-3-methylimidazolium chloride (EMIC) and aluminum chloride (AlCl_3) was carried out in a two-electrode setup (i.e., cathode and anode) at room temperature, with a current density of 20 mA/cm². Secondly, the container specimen with a ~20 μm thick Al coating was heat treated at 700 °C for 8 h in air, to form an aluminized coating. Finally, the aluminized coating on the container was ground, and then artificially oxidized at 700 °C for 50 h in argon gas stream (purity: 99.99%) with 20–30 ppm O₂, to form the surface Al_2O_3 scale.

2.2. Permeation installation and procedures

A schematic of T-permeation experimental installation is shown in Fig. 1. This system is an all-metal vacuum system, including an ion chamber (IC), a diffusion cell (DC), a heater (HT) and its control system, a T-source gas bottle and an argon supply system. Except for the Ar supply system, all parts are located in a glove box connected to a vent detritiation system (VDS).

The bare and coated specimens were separately welded into a diffusion cell made of 321 type stainless steel (inner diameter: $\Phi 95$ mm, length: 160 mm, thickness: 3 mm). As shown in Fig. 1, there was a gap between the inner surface of the diffusion cell and the outer surface of the specimen through which the Ar-gas was supplied to sweep the permeated tritium to the ion chamber. All gas supply tubes were of 316L stainless steels affixed electric heat tapes which can heat to 110 °C for surface cleaning. All valves and fittings were selected and fabricated to minimize tritium losses. Joints were welded, and metal ring gaskets were used where welding was not possible. The specimen and Ar sweep gas were heated using furnaces (HT1 and HT2 in Fig. 1). The temperatures could be set up to 800 °C by temperature controllers, with thermocouples, allowing a temperature stability of ± 5 °C. Two thermocouples positioned along the outer surface of the diffusion cell determined the heating power and temperature distribution of HT2, and one thermocouple inserted in the diffusion cell with its head touching the specimen determined the specimen temperature.

The procedure of tritium permeation measurements using the installation shown in Fig. 1 was described as follows. Before the experiment initiation, the installation, accompanied by the diffusion cell annealed at 120 °C, was continuously purged by the Ar sweeping gas (purity: 99.999%) along the arrow direction to clean the surface of gas lines, as shown in Fig. 1. The system was conditioned until the signal of the ionization chamber became stable, and then the tritium gas (purity: 99.9%) was poured into the container specimen within the diffusion cell. Then, with the Ar-gas sweeping over the outer surface of the specimen, the diffusion cell

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