Fusion Engineering and Design 102 (2016) 94–98

Contents lists available at ScienceDirect

Fusion Engineering and Design

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

Performances of AlN coatings as hydrogen isotopes permeation barriers

Jie Wang, Qun Li, Qing-Yun Xiang, Jiang-Li Cao*

Beijing Key Laboratory for Corrosion–Erosion and Surface Technology, Institute of Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China

HIGHLIGHTS

- The D-PRF of AlN-HIPB was studied.
- The morphologies of AlN-HIPB remained compact after deuterium permeation tests.
- The diffraction peaks maintained stable.
- The nano-hardness increased after the deuterium permeation tests.

ARTICLE INFO

Article history:
Received 30 May 2015
Received in revised form
25 November 2015
Accepted 25 November 2015
Available online 17 December 2015

Keywords:
Thermonuclear fusion
Hydrogen isotope permeation barriers
AlN coating
Deuterium permeation

ABSTRACT

Hydrogen isotope permeation barriers (HIPB) have great potential applications in the fields of hydrogen energy and thermonuclear fusion. In this study, the AlN-HIPB were prepared on 316 L stainless steel by RF magnetron sputtering. The properties of AlN-HIPB were studied, including the deuterium permeation reduced factor (D-PRF), structures and nano-hardness. The D-PRF of 0.4 μm AlN-HIPB could reach 36 at 600 °C, and gradually rise with decreasing permeation temperature. The D-PRF reached 53 at 400 °C and 144 at 250 °C, respectively. The coatings remained dense and the grains were spherical after the deuterium permeation test. The AlN (1 0 0) diffraction peaks appeared and maintained stable during the deuterium permeation process. The nano-hardness of the coatings increased from 5.96 GPa to 7.41 GPa and the elasticity modulus also increased from 156.6 GPa to 210.6 GPa after the deuterium permeation.

1. Introduction

Because thermonuclear fusion energy and hydrogen energy are clean and economical, they are considered to be alternative energy of fossil energy [1]. Thermonuclear fusion reactor systems are fuelled by hydrogen isotopes. The hydrogen isotopes can easily permeate through construction materials because of small atomic radius, which may result in embrittlement of constructional materials, energy waste and radioactive contamination [2–6]. Therefore, it's of great scientific and practical value to reduce the hydrogen isotopes permeation. The international solution method is to make hydrogen isotopes barriers in the inner wall of constructional materials.

With the development of international thermonuclear fusion reactor (ITER), studies on hydrogen isotopes permeation barriers have been widely carried out [7–12]. The aluminide coatings were selected as promising candidate material for HIPB owing to high melting point, high strength, high chemical stability and hydrogen isotope permeation reduction factor [13–16]. For example, Zhang et al. prepared the aluminium rich coating on HR-2 steel, which can reduce deuterium permeation by 2–3 orders of magnitude [17]. He et al. deposited Al_2O_3/Cr_2O_3 composite film on 316 L stainless steel by metal organic chemical vapour deposition (MOCVD). Their composite films can significantly reduce the deuterium permeation [18]. So far, the studies available are mainly focused on aluminium oxide and its composite coatings [19–25]. However, the aluminium nitride coatings used as HIPB have hardly been reported in previous studies. Aluminium nitride has excellent physical and chemical properties such as high hardness, corrosion resistance, mechanical strength, high electrical resistivity and thermal stability [26], which are advantageous for HIPB use in harsh fusion environments. The AlN not only has 3000 °C melting point [27] but also remains stable in high temperature reducing atmospheres. It is necessary for HIPB to have a high resistivity which can significantly reduce the magneto-hydrodynamic (MHD) effect. The resistivity of AlN is over 10^{13} Ω cm and could meet the demand of HIPB [28]. AlN has

* Corresponding author.
E-mail address: perov@sina.com (J.-L. Cao).

http://dx.doi.org/10.1016/j.fusengdes.2015.11.043
0920-3796/

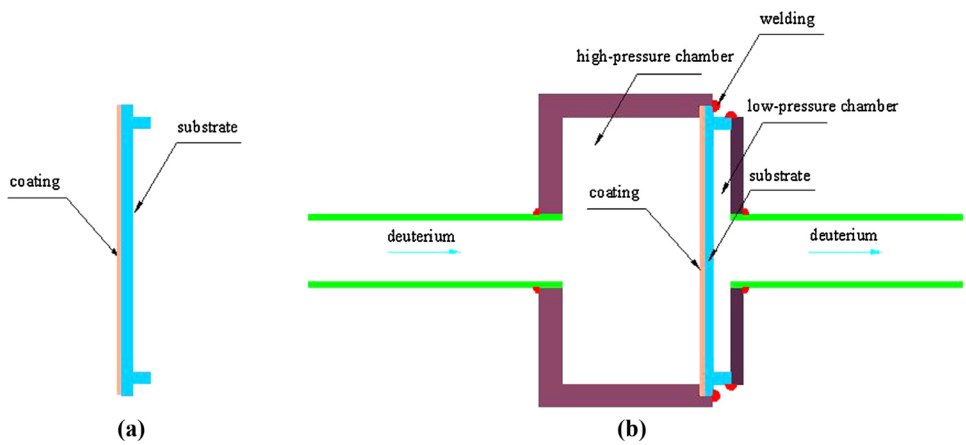

Fig. 1. Diagram of the deuterium permeation sample. (a) Wafers used for depositing coatings. (b) The permeation chambers.

a hexagonal crystal structure [27]. The Al atoms and N atoms combine each other by covalent bonds. The strong Al–N covalent bonds may hold the aluminum atom and nitrogen atom together firmly, which lead to higher activation energy of diffusion for deuterium atoms to overcome in AlN coatings than that in metals. AlN is stable in lithium with low nitrogen concentrations at temperatures of interest [29,30]. AlN coatings exhibit good compatibility with Li containing specific additions of Al and N [28].

In this paper, the AlN-HIPB were prepared on 316 L SS by RF magnetron sputtering and their abilities of impeding the hydrogen isotopes permeation were discussed. The micro-morphologies, structure and nano-hardness of the AlN-HIPB after deuterium permeation were also investigated systematically.

2. Experimental

2.1. Sample preparation

First, the 316 L SS was cut into small wafer with 25 mm diameter and 0.5 mm thickness. Then, the small wafers were ground with 2000 grid water sand paper and cleaned with an ultrasonic cleaner at 50 °C according to the following order: 20 min in sodium hydroxide solution, 20 min in deionized water and 20 min in alcohol. Second, the AlN-HIPB were deposited by the radio frequency reactive magnetron sputtering. The target was metal aluminum with a purity of 99.99%. The target-substrate distance was 60 mm. The temperature of the substrate was 300 °C. The chamber was vacuumed to lower than 8×10^{-4} Pa and then argon with a purity of 99.999% and nitrogen with a purity of 99.999% were introduced into the chamber. The total pressure was kept at 1.2 Pa and the flux ratio of argon to nitrogen was 3:1. After 4 h deposition, the samples were took out and were annealed in nitrogen at 600 °C for 1 h to accelerate the crystallization and diffusion between the AlN-HIPB and the substrate.

2.2. PRF measurement

One of the main parameters of the HIPB is permeation reduction factor (PRF), which is used to characterize the ability of the coatings impeding hydrogen isotopes permeation. PRF is generally defined as the ratios of the ion current of deuterium permeating through the coated samples to that of deuterium permeating through uncoated samples. The larger the PRF is, the better the impeding ability of the coating possesses. Deuterium is selected as permeation gas because of the radioactivity of tritium and high abundance of hydrogen which results in the high ion current background. The HIPB samples were connected into its gas circuit by laser welding in order to protect the coatings from the welding heat. The descriptions of laser welding and samples were shown in Fig. 1. The coatings are prepared on the left side of the wafer. The deuterium from the high-pressure chamber permeated through the samples and released on the low-pressure chamber. The laser welding technology used is pulse laser welding. The red points mean the laser welding areas. As shown in Fig. 1(b), there were enough superfluous space around the sample which are prepared for laser welding.

An experimental facility was set up to measure the PRF of the coatings called the Gas Permeation Measuring Instrument similar to the equipment in [31]. The samples divide the facility into two parts, the high-pressure chamber and low-pressure chamber. One side of the samples are exposed to deuterium which is defined as high-pressure chamber. The gas permeated through the samples and released on the other side which is defined as low-pressure chamber. Before the measurement, the leakage check was carried out to ensure the accuracy of data and safety of the measurement by charging gas into the measurement system. On the other hand, the ion currents of gas such as CO_2, N_2 and H_2O were also monitored in the measurement process. The intense change of these ion currents could indicate the leakage of the measuring system. When the inner pressure of the instrument was lower than 1×10^{-5} Pa, the high pressure deuterium was introduced into the high-pressure chamber. The samples were heated by a resistance furnace whose temperature could change in certain ranges. The ion current of deuterium permeating through into the low-pressure chamber could be measured by the quadrupole mass spectrometer. Several deuterium permeation tests had been carried out to ensure the accuracy of the experimental data.

In order to clarify the impacts of vacuum annealing and deuterium permeation on AlN-HIPB, the whole deuterium permeation were divided into two steps: 600 °C vacuum annealing process for 24 h and deuterium permeation.

2.3. Other characterization

The microstructure of the AlN-HIPB were examined by scanning electron microscopy (SEM, ZEISS SUPRA 55), the phase composition of the coatings was characterized by X-ray diffraction (XRD, RIGAKU D/MAX-RB). A nano-indenter (Nano Indenter, AGILENT XP) were utilized to measure the nano-hardness and the elasticity modulus of the coatings.

3. Results and discussion

Fig. 2(a) and (b) shows the morphologies of surface and cross-section of as-deposited AlN-HIPB. It is observed from Fig. 2(a) that