



Preparation of layered α -Al₂O₃/TiO₂ composite coating by pack cementation process and subsequent thermochemical treatment

Yan Wang, Shuaijie Feng, Dawei Liu, Cheng Zhang, Juexin Xu, Chunlan Luo, Jinping Suo*

State Key Laboratory of Material Processing and Die & Mould Technology, School of Materials Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, PR China

ARTICLE INFO

Keywords:

Al-Ti coatings
 α -Al₂O₃/TiO₂ composite coating
 Pack cementation
 Thermochemical treatment
 Tritium permeation barrier

ABSTRACT

An Al-Ti coating deposited by pack cementation technique was subjected to the thermochemical treatment. The Al-Ti coating consisted of an outer AlTi-rich layer and an inner transition layer. After the direct oxidation treatment, the coating surface was covered by many particle agglomerations and the scale was porous. A 10 μ m thick oxide layer, which consisted of an outer TiO₂ dominated layer and an inner α -Al₂O₃ dominated layer, had grown on top of the coating. The multilayer structure of this coating was “TiO₂/ α -Al₂O₃/transition layer”.

In order to prepare a better α -Al₂O₃/TiO₂ composite coating, nitridation pretreatment was added before oxidation treatment. The surface of resulting coating was compact and had no obvious particle agglomerations. The outer oxide layer, with a thickness of approximately 4 μ m, exhibited a bilayer structure with an outer α -Al₂O₃ dominated layer and an inner TiO₂ dominated layer. The multilayer structure of this coating was “ α -Al₂O₃/TiO₂/transition layer”. Beneath the oxide scale, a thin and continuous nitride layer was present, slowing down oxygen transport into the coating significantly. The oxidation behavior of Al-Ti coating changed with the nitridation pretreatment, and a better α -Al₂O₃/TiO₂ composite coating was obtained.

1. Introduction

Deuterium–tritium fusion reactors have been widely studied for several decades because of its capacity to produce exhaustless energy [1]. One of the most crucial issues for the development of fusion reactors is tritium permeation through structural materials in blanket systems accompanied by fuel loss and radiological hazards [2]. Meanwhile, the hydrogen absorption of the structural steels will result in hydrogen embrittlement and a subsequent degradation of the mechanical properties [3]. Application of tritium permeation barriers (TPBs) is a promising method to solve this problem. Some attempts to produce a reliable barrier material and deposit it on structural materials have been reported in recent years. Ceramic tritium permeation barriers have been proved to be a very effective way [4] to decrease tritium penetration. As demonstrated in the literature, the usual TPBs include α -Al₂O₃ [1], TiO₂ [5], TiN [6], TiN/TiC [7], Cr₂O₃ [8], Er₂O₃ [9] and SiC [10].

Among these ceramic coatings, the layer of alpha alumina appears to be the most promising candidate for a TPB due to its excellent property of hydrogen permeation resistance, good compatibility with liquid PbLi, self-healing ability and excellent stability against neutron irradiation [11,12]. Levchuk et al. [12] reported that the alpha alumina

with only 1 μ m thick exhibited a deuterium permeation reduction factor of 1000 in the temperature range of 973–1073 K. Several methods of preparing α -Al₂O₃ coatings on structural materials have been attempted, such as hot dip, vacuum plasma spray, pack cementation and chemical vapor deposition, etc. Nevertheless, a high demand is required for the formation of crystalline alumina [13,14]. It is difficult to form α -Al₂O₃ thin films because the high temperature about 1200 °C is required [15]. In addition, it is still challenging to prepare coatings without cracks and pores, control the thickness of the coatings and improve the adhesion between the coating and the substrate [1]. The thermal expansion coefficient of α -Al₂O₃ ceramic coating is very different from that of the substrate [16,17], which often results in the formation of pores and cracks at interfaces at high temperature, leading to the failure of the coatings [18–20]. As a consequence, it is of great value to study new preparation methods to obtain compact and uniform α -Al₂O₃-based ceramic coatings as TPBs.

In this study, a new combined process including pack cementation and thermochemical treatment was utilized to prepare α -Al₂O₃/TiO₂ composite coating as TPBs. On the one hand, we hope to realize the formation of α -Al₂O₃-based ceramic coatings as TPBs at lower temperature. On the other hand, we expect the multi-interface film structures show more effective permeation suppression for promising TPBs

* Corresponding author.

E-mail address: jinpingsuo@hust.edu.cn (J. Suo).

Table 1
Chemical composition of the SCRAM steel in wt%.

Element	Si	P	Ti	V	Cr	Mn
Concentration (wt%)	0.083	0.0073	0.015	0.25	9.27	0.46
Element	W	C	S	N/ppm	Fe	
Concentration (wt%)	2.34	0.097	0.0016	700	Balance	

application [21,22]. The combination of pack cementation and thermochemical treatment not only can be easily applied to prepare coatings for large surfaces and complex geometry shapes in fusion reactors, but also can control the coating thickness and improve the bonding strength between coating and substrate. The purpose of this study was to provide a new technology as a reference for the preparation of ceramic coatings, which can be applied not only to the preparation of TPBs but also to the preparation of wear and corrosion resistance coatings in the field of engineering.

2. Experimental

2.1. Sample preparation

In this investigation, the codeposition of Al and Ti was carried out on super-clean reduced activation martensitic (SCRAM) steel. Table 1 shows the chemical composition of this steel. The diameter of the steel rod was 20 mm. It was cut into 5 mm thick discs, of which the surfaces were ground and polished to a 1000-grit finish. The samples were degreased in acetone and cleaned with deionized water by ultrasonic cleaning instrument before coating preparation process.

The preparation of the coating was carried out simultaneously with the heat-treatment of the substrate steels for fear that the mechanical properties of steels were affected by the high temperature. It is known from our early research that the microstructure of SCRAM steel can be remarkably refined by the twice-quenching treatment, contributing to the decrease of its ductile brittle transition temperature (DBTT) [23]. In this study, samples went through the twice-quenching treatment: 1020 °C 0.5 h quenching/760 °C 2 h tempering + 1000 °C 0.5 h quenching/760 °C 2 h tempering. Codeposition of Al and Ti on substrate took place simultaneously with the first quenching of the steel. Oxidation treatment or nitrogen treatment of the coatings was carried out while the steel was first tempered. Second quenching and tempering were conducted while the coatings were further oxidized.

2.2. Pack cementation

Codeposition of Al and Ti on SCRAM steel was conducted by pack cementation process. Deposition conditions (e.g., substrate roughness, temperature, and deposition time) in previous studies [22] were used to ensure high surface coverage of the coating without stripping. Pack powder mixtures consisted of Al and Ti as a depositing source, a halide salt ammonium chloride as an activator and Al₂O₃ powders as the inert filler. Pack was prepared by burying the substrate in a well-mixed pack powder mixture in a cylindrical alumina crucible with a 36 mm diameter and 50 mm height, which was then sealed with alumina lid and high-temperature cements. The crucible was placed into an electric muffle furnace after the cement sealants cured. The temperature was raised to 1020 °C at a heating rate of 7 °C·min⁻¹ and sustained for a required period. At this high temperature, a series of halide vapors could form primarily due to the reaction of activator with the depositing elements. Then they would permeate into the porous pack to reach the substrate surface, on which decomposition of the halide vapors would happen. With further solid state diffusion between the deposited

Table 2
The pack powder composition and the holding temperature/time.

Powder mixture composition, wt%				T/°C	t/h
Al	Ti	Al ₂ O ₃	NH ₄ Cl		
19	38	40	3	1020	0.5

elements and the substrate elements, a diffusion coating formed at last [24–26]. Table 2 shows the pack composition and the holding temperature/time. After the codeposition process was completed, the crucible was taken out from the furnace and cooled to room temperature in water. The coated sample was then removed from the crucible and ultrasonically cleaned in ethanol to remove any embedded packaging material.

2.3. Thermochemical treatment

To obtain a fine performance α -Al₂O₃/TiO₂ composite coating, the Al-Ti coating was then nitrated and oxidized. Nitridation treatment of the coating was carried out simultaneously with first tempering of the substrate steel, so this process was held at 760 °C for 2 h. In order to ensure that the surface of Al-Ti coating could be fully nitrated, a relatively large ammonia flow rate was selected during the nitridation process, which was 200 ml/min. The subsequent oxidation process was conducted in air while second quenching and tempering of the substrate were carried out.

Meanwhile, the Al-Ti coating was subjected to another treatment: the coating was oxidized directly in air while first tempering, second quenching and tempering of the substrate were conducted. Details of the treatment conditions of the specimens are given in Table 3.

2.4. Characterizations

The phases in the coating were characterized by the X-ray diffraction (XRD) with CuK α radiation at 40 kV. The surfaces and cross sections of the coatings were observed with a scanning electron microscopy (SEM). The chemical compositions of the coated specimens were analyzed using energy dispersive spectroscopy (EDS) coupled to SEM.

3. Results

Fig. 1 shows the surface microstructure of a specimen, on which surface an aluminum titanium layer was deposited. The structure was fine and uniform, suggesting that the Al-Ti coating had a grain growth morphology similar to that of the CVD.

Fig. 2 presents XRD spectrums measured from the coating surfaces. It can be observed that the XRD spectrum of Al-Ti coating (Fig. 2a) is sophisticated, indicating the presence of multiple phases in the coating surface. If assuming that there was no new or unknown phase, the XRD result showed that the phases of the coating surface included Al₃Ti, AlTi, Ti and Fe₂Ti. At this stage, it is impossible to estimate the proportion of each phase in the coating surface. However, the EDS data shown in Fig. 1b indicated that the main phase of the top surface was AlTi.

Table 3
Thermochemical treatment conditions for Al-Ti coating.

Specimen	Material	Thermochemical treatment conditions
C1	Al-Ti coating	Oxidizing/760 °C/2 h/air-cooling + oxidizing/1000 °C/0.5 h/air-cooling/760 °C/2 h/air-cooling
C2	Al-Ti coating	Nitriding/760 °C/2 h/air-cooling
C3	Al-Ti coating	Nitriding/760 °C/2 h/air-cooling + oxidizing/1000 °C/0.5 h/air-cooling/760 °C/2 h/air-cooling

Download English Version:

<https://daneshyari.com/en/article/5464402>

Download Persian Version:

<https://daneshyari.com/article/5464402>

[Daneshyari.com](https://daneshyari.com)