



Research Paper

Growth of inter-metallic compound layers on CLAM steel by HDA and preparation of permeation barrier by oxidation



Jun Chen^a, Xianfen Li^{a,*}, Peng Hua^{a,*}, Chunyan Wang^a, Ke Chen^a, Yucheng Wu^a, Wei Zhou^{a,b}

^a School of Materials Science and Engineering, Hefei University of Technology, Hefei 230009, China

^b School of Mechanical and Aerospace Engineering, Nanyang Technological University, Singapore 639798, Singapore

ARTICLE INFO

Keywords:

CLAM steel
HDA process
IMC layers
Heat-treatment
Al₂O₃ film

ABSTRACT

Hot-dip aluminizing (HDA) on China Low Activation Martensitic (CLAM) steel and followed by oxidation to obtain an Al₂O₃/Fe_xAl_y layer covering was considered to be a promising method to resist the harsh environments in fusion reactors. In the present study, CLAM steel was coated in molten pure Al and Al-0.3Ce (wt.%) melt. The growth behavior of the inter-metallic compound (IMC) layers after different immersion time and temperature was investigated and described. The IMC layer mainly consists of Fe₂Al₅ and FeAl₃ after hot-dipping. And the addition of Ce in the molten pure Al could promote the growth of IMC layers, which provided the idea of obtaining a certain thickness of the IMC layer at a lower temperature. After two independent oxidation processes (normalized heat-treatment and 760 °C for 15 h) in air, the Fe₂Al₅ and FeAl₃ of IMC layers had been transformed into ductile phases (FeAl₂ and FeAl), and Al₂O₃ layers were detected on the coating surface. The oxidation of 760 °C for 15 h was considered to form a denser Al₂O₃ film. However, cracks and pores were observed in the IMC layer after oxidation due to the heat-treatment and cooling methods

1. Introduction

Reduced Activation Ferritic/Martensitic (RAFM) is considered as the structural material in the International Thermonuclear Experimental Reactor (ITER) Test Blanket Systems (TBS) [1,2]. Quite a lot of steels (9Cr-2WVTa, F82H, JLF-1, ARAA and India RAFM steel) have been performed as RAFM steels. Simultaneously, CLAM steel is selected as one of the primary promising candidate of structural material for the ITER [3–5]. Liquid Pb-Li cooling blankets module is one of TBS which plays an important role in facing lots of performance and operational issues (POIS) such as Pb-Li corrosion, tritium permeation, tritium leakage and magnetohydrodynamics effects [6–8]. Compatibility testing of corrosion was performed in flowing Pb-15.7Li at flow velocity of 0.22 m/s with 480 °C and 550 °C, respectively [9–11], a fairly rapid rate of corrosion about 400 μm/s occurred at the higher temperature of 550 °C. Hence, anti-corrosion research is necessary and urgent. The technology of coating on matrix material as permeation barriers seems to be a promising method [12,13]. A lot of plating processes and plating materials have been performed to find a fine way to solve these POIS, such as forming metallic (Sn) layer [14], non-metallic (N, Si) layer [15,16], ceramic (TiO₂, Y₂O₃, SiO₂, CrO₃, Al₂O₃ etc.) layer [17–24]. The techniques of HDA, plasma spray, chemical vapor deposition (CVD), toluene-based electrolytes (ECA) and ionic

liquids (ECX) process (subsequent heat treatment) have been used to obtain a wide variety of coatings [21,25–31]. The coatings fabricated by ECA and ECX process have been tested to prove their compatibility to Pb-Li [30,31]. HDA is a commonly used technology to plate technology [25,32,33] and the HDA process offers a good possibility to produce aluminide coatings by pumping liquid aluminum through the whole blanket to form the required layer on the surfaces of complex-shaped components. Although the coating obtained by the HDA process may be high on the Al-content, the coating can withstand the corrosion attack in flowing Pb-15.7Li even at high flow velocities [34]. The Al₂O₃ coating would be considered in all the coolant channels inside blanket and all the pipes in the auxiliary system to be the permeation barriers [35]. The overall performance such as corrosion resistance, decorative and antioxidant properties has been improved because of the plating aluminum on the matrix material. Moreover, the coating surface will naturally generate a layer of Al₂O₃/Fe_xAl_y film, so that corrosion resistance and high temperature oxidation resistance have been greatly improved.

Fe can react with Al to form Fe-Al inter-metallic compounds (Fe₃Al, FeAl, FeAl₂, Fe₂Al₅ and FeAl₃ phases) at an equilibrium state. It is generally believed that the coating is divided into two layers, the outer aluminum layer with the same composition of Al melt and IMC layer mainly composed of Fe₂Al₅ phase. The composition and thickness of the

* Corresponding authors.

E-mail addresses: lxftt@163.com (X. Li), weldinghua@163.com (P. Hua).

Table 1
Parameters for the experiments under study.

Melt composition		Immersion temperature T (°C)	Immersion time t (min)
Pure Al	Al-0.3Ce (wt. %)		
✓	✓	710	4
✓	–	760	0.5
✓	–	760	2
✓	✓	760	4
✓	–	760	6
✓	–	760	8
✓	–	760	10
✓	–	760	15
✓	✓	810	4
✓	✓	860	4
✓	–	910	4

IMC layer is also affected by the composition of aluminum liquid, matrix composition and organization [36–38]. In this paper, $\text{Al}_2\text{O}_3/\text{Fe}_x\text{Al}_y$ coating on CLAM steel substrates had been prepared with HDA process and followed by oxidation. To acquire good integrated coating performance which can be reflected in cracks, growth stress, adhesion and porosity of the interface, the rare earth element (Ce) was added into the coating [39,40]. In addition, the addition of rare earth (Ce) can change the diffusion kinetics of the oxidation process of the alloy to change the formation and growth mechanism of oxide film, promote the formation of steady-state Al_2O_3 phase at relatively low temperature [41–43]. Optical microscopy (OM), scanning electron microscopy (SEM) observation, energy dispersive spectrometer (EDS), X-ray diffraction (XRD) testing for the specimens had been carried out after experiment to evaluate the properties of the coating.

2. Experimental

2.1. Specimen preparation

CLAM steel was used as the matrix material to dip into the Al melt (pure aluminum with a purity of 99.99%) and Al-0.3Ce melt under different condition, as shown in Table 1. The chemical composition of CLAM steel produced by IMR (Institute of metal research, Chinese Academy of Sciences) in this study is: 8.93Cr, 1.43W, 0.48Mn, 0.19V, 0.10Ta, 0.05Si, and 0.09C in wt.%, and Fe for the balance. The matrix samples ($20 \times 15 \times 2.5 \text{ mm}^3$) with a small hole (fix the specimens during the HDA process) on the surface were processed by wire cutting. The rectangular specimens were normalized heat-treated (NHT) at 980 °C for 30 min followed by cooling in air to room temperature (RT) and then tempered at 760 °C for 90 min followed by air cooling to RT.

The detailed heat treatment conditions and composition for CLAM steels can be found in Refs. [44–47]. The surface of the specimens had been grinded on 600–1200 emery papers and then polished carefully to obtain mirror face using diamond abrasive paste with granularity of 2.5 μm . Subsequently, the specimens were treated by 15 vol.% HCl water solution to remove the native oxide film and improve the wettability before the HDA process. The Al-0.3Ce melt was chosen to investigate the influence of Ce on the IMC layers. Ce was added into the aluminum melt at 760 °C, and then the melt was stirred periodically to ensure the two elements being fully melted and homogenized. All the melt were covered by a layer of KCl-NaCl powder as a covering agent to prevent the metal from being oxidized at high temperatures and play a role of insulation. A uniform coating layer on each specimen could be observed after HDA process. Vicker's microhardness tests were carried out with an applied load of 300 gf for 10 s. X-ray diffraction was measured by D/MAX2500 V from 10° to 90° with a scanning speed of 5° per minute.

2.2. Oxidation technique

An Al/Fe-Al layer has been formed with compositional gradient on the surface of CLAM steel after HDA process. Just relying on this layer as a barrier is far away from the requirements. Oxidation of Al/Fe-Al layer is necessary to form a protective Al_2O_3 film in order to meet the requirements of permeation barrier. Specimens after HDA were ultrasonically cleaned in acetone for 15 min, dried, and then placed in alumina crucibles which were heated to the heat-treatment temperature in a muffle furnace. Subsequently, two oxidation methods, NHT process (980 °C for 30 min, air-cooled; 760 °C for 90 min, air-cooled) and 760 °C for 15 h, were carried out to transform the brittle Fe_2Al_5 layer into softer phases and form a high quality Al_2O_3 film on the surface.

3. Results and discussion

3.1. Effect of immersion time and temperature on the coatings in pure Al melt

Typical layer micrographs for the cross-section of specimens dipped in pure Al melt are shown in Fig. 1 with different temperature (T) and time (t).

As we can see from these images, each microstructure was divided into three distinct regions, the outer Al layer, the intermediate IMC layer, and the inner CLAM steel matrix layer. From the Al side, the main Fe_2Al_5 phase was demonstrated as reported by Refs. [36–38]. Tongue-like phases (i.e. IMC layers) were formed because of Fe concentration at the interface and reaction with Al. Layers of various thicknesses were formed on the substrate surface which were tightly connected with the

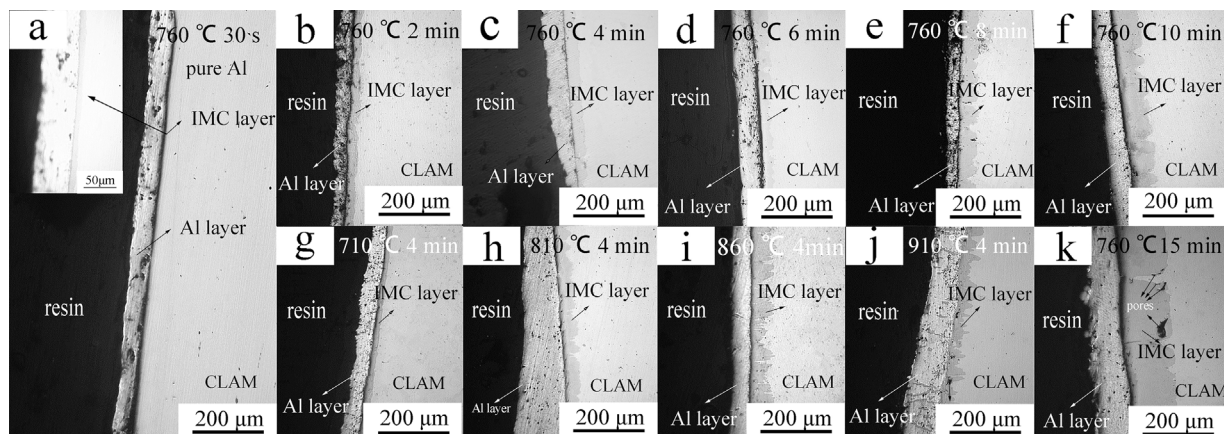


Fig. 1. OM images of cross-section for the Fe/Al diffusion couple in pure Al melt.

Download English Version:

<https://daneshyari.com/en/article/6743719>

Download Persian Version:

<https://daneshyari.com/article/6743719>

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