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Microstructure and peeling behavior of MOCVD processed oxide insulator coating before and after ion beam irradiation

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ABSTRACT

The mechanical durability and soundness of the several oxide coating materials used as the electrical insulator and tritium permeation barrier are important design parameters on an advanced liquid breeding blanket system. We tried to investigate the adhesion strength evaluation due to the peeling behavior on the MOCVD processed multilayered oxide coating (Er_2O_3/Y_2O_3) on the stainless steel (SUS) substrate before and after Cu^{2+} ion beam irradiation using the nano-scratch tester. The adhesion strength of the coating material was able to estimate easily from the scratch trace and scratch stress, and the nano-scratch test was suitable method to evaluate the mechanical durability and soundness. After the Cu^{2+} ion beam irradiation, the adhesion strength was decreased with increasing the displacement per atom (dpa). The adhesion strength degradation by the Cu^{2+} ion beam irradiation was caused by the embrittlement of the thicker and amorphous Fe-(Y)-O interlayer formation between Y_2O_3 buffer layer and SUS substrate based on the displacement damage dose effect.

1. Introduction

There are many key issues and technologies to realize some advanced breeding blanket systems using liquid metals (Li and Pb-Li) and molten-salts (FLiBe and/or FLiNaBe). In particular, development of the both electrical insulator and tritium permeation barrier components is the critical key issue to satisfy the technical specification and engineering design on these advanced blanket systems. Erbium oxide (Er_2O_3) is well known as the one of the candidate oxide coating materials for the electrical insulator and tritium permeation barrier due to the higher electrical resistivity at high temperature, high compatibility with liquid Li and significant suppression of the tritium leakage [1,2]. We have, therefore, been developed for Er_2O_3 coating process on broad and complicated shaped components, which were blanket structures and duct pipes, from the viewpoint of the real blanket system production. Recently, we demonstrated to form Er_2O_3 coating into the stainless-steel pipe interiors using metal organic vapor deposition (MOCVD) process [3–5]. In addition, we succeeded to form a thicker and oriented MOCVD Er_2O_3 coating with high crystallinity by the double stacked layer formation using an intermediate buffer layer such as yttrium oxide (Y_2O_3) and cerium oxide (CeO_2) [6,7]. In this way, the progress of the several Er_2O_3 coating developments including the fabricate process is steadily performed.

On the several coating materials as the electrical insulator and tritium permeation barrier components on advanced breeding blanket, it apprehended that the mechanical properties of the coating material were deteriorated by the high-temperature and heavy neutron irradiation. In particular, neutron irradiation effect was the most important factor to predict the fracture and life durations of the coating material. However, there is not the 14 MeV fusion neutron and/or thermal neuron irradiation facility in worldwide. Therefore, the simulated irradiation experiment such as the heavy ion beam irradiation would mainly use to investigate the fusion neutron irradiation effect. The heavy ion beam irradiation experiment using the accelerator would be equivalent to acceleration irradiation compared with nuclear fusion reactor environment, and it could gather the 14 MeV neutron irradiation effect on microstructure and mechanical property of the fusion component materials.

On the other hand, we thought that the establishment of the systematic mechanical evaluation method on the electrical insulator and tritium permeation barrier coatings was the most important issue to decide the technical specification and engineering design on an advanced breeding blanket. Therefore, we approached to evaluate the adhesion strength, which was one of the important mechanical properties on the coating materials, before and after heavy ion beam irradiation. In the future, to clarify the adhesion strengths of the coating

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material under the various blanket operation conditions will contribute to the blanket design by providing estimate of the soundness, the product lifetime and the durability of the coating material.

In generally, there are a few methods to evaluate the adhesion strength of the various soft coating materials such as the metal-plate layer and polymer film, and they are so-called the cross-cut method and pull off method [8,9]. These methods are theoretically simple and easy technique, but these are not suitable for the hard and multi-stacked coating materials. In cases of the cross-cut and pull off methods, it was very difficult to make small grid-shape slit in the hard coating and to quantify quantitatively the adhesion strength. On the other hand, the scratch method was made applicable to the case which needs much larger external forces to separate from a substrate like the hard and multi-stacked coatings such as car-paint and diamond like carbon (DLC) thin coating [10]. Furthermore, the adhesion strength evaluation of the several hard coatings by the scratch method was determined by the Japan Industrial Standard (JIS) [11]. We thought that the nano-scrtach measurement would become one of the useful mechanical adhesion evaluation method to build up the dynamic data-base of the oxide coating as the fusion component.

In this study, the adhesion strength measurement of MOCVD processed Er_2O_3/Y_2O_3 buffer multi-stacked coating on the stainless steel (SUS316) substrate before and after the Cu^{2+} ion beam irradiation at room temperature was carried out using the nano-scratch method. Effects of the Cu^{2+} ion beam irradiation on microstructure and adhesion strength of Er_2O_3/Y_2O_3 multi-stacked coating on the SUS 316 substrate were investigated.

2. Experimental procedures

2.1. Preparation and Cu^{2+} ion beam irradiation at room temperature on the MOCVD processed Er_2O_3/Y_2O_3 buffer multi-layered coating samples

We used the stainless steel 316 (SUS316) plate having 17 mm in diameter as the metal substrate. The surface of raw SUS316 substrate was polished using several emery papers (#400-#1200) and mirror finish buffing with alumina abrasives having 0.3 and 0.05 µm in a diameter. This polishing was carried out before Y_2O_3 buffer layer formation via the Radio frequency-Physical Vapor Deposition (RF-PVD) process. The yttrium oxide (Y_2O_3) was selected as the buffer oxide material in this study because the lattice constant of Y_2O_3 was extremely similar to the Er_2O_3 crystal. The thickness of Y_2O_3 buffer layers was adjusted to approximately 500 nm. And Er_2O_3 layer was formed on the Y_2O_3/SUS316 substrate by the MOCVD process. The detail MOCVD conditions, achieved by configuration of the MOCVD apparatus, metal organic material and typical oxygen and argon gas flow rates, were already described [3–6]. The Y_2O_3/SUS316 substrates were placed into the plateau temperature region of the MOCVD reactor. The deposition condition was 550 ^{0}C for 3 h.

After the Er_2O_3 deposition on the Y_2O_3/SUS substrate, the nanostructure of the multi-stacked deposition layer was observed by field-emission typed scanning electron microscopy (FE-SEM; JSM-7800F) and scanning transmission electron microscopy (STEM; JEM-2800). Typical TEM image of the cross-sectional area in Er_2O_3/Y_2O_3/SUS316 regions after MOCVD process is shown in Fig. 1. The thickness of Er_2O_3 and Y_2O_3 layers in all MOCVD processed samples were obtained to be approximately 800 nm and 500 nm, respectively.

The Cu^{2+} ion beam irradiation in this study carried out using the tandem typed accelerator at Research Institute for Applied Mechanics (RIAM), Kyushu University. Er_2O_3/Y_2O_3/SUS316 multi-layered coating sample was set into the vacuum chamber having under 10^{-4} Pa, and the Cu^{2+} ion beam having 2.4 MeV was irradiated to the Er_2O_3 layer surface. Depth profile of the displacement rate was calculated by the SRIM code with full-cascade options. A simulated depth profile of the displacement rate on the Er_2O_3/Y_2O_3/SUS316 multi-layered coating sample is shown in Fig. 2. The nominal displacement rate was defined

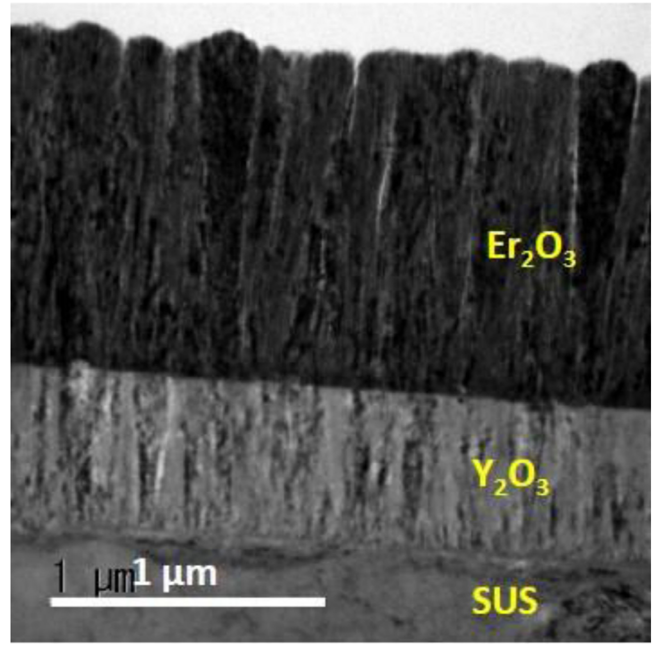

Fig. 1. Typical TEM image of the cross-sectional area in Er_2O_3/Y_2O_3/SUS316 regions after MOCVD process.

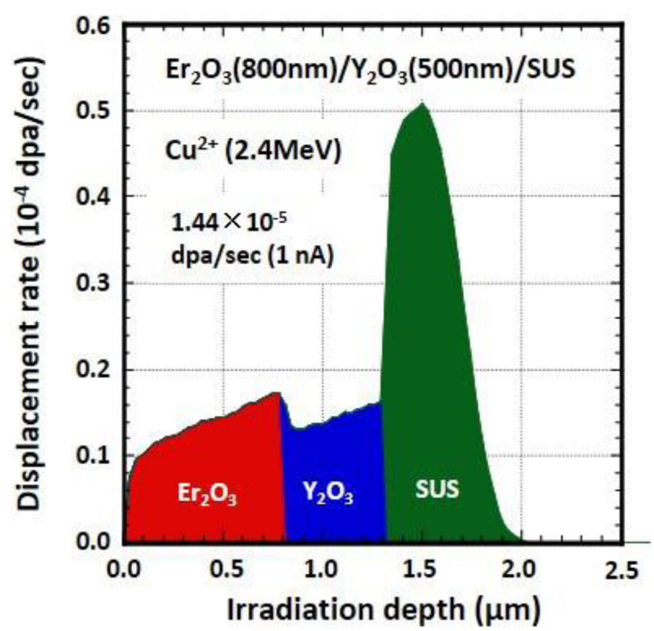

Fig. 2. The simulated depth profile of the displacement rate of the Cu^{2+} ion beam (2.4 MeV) on the Er_2O_3/Y_2O_3/SUS316 multi-layered coating sample calculated by the SRIM code with full-cascade options.

as 1.44×10^{-5} dpa/sec at the depth of 480 nm from the surface, and this rate corresponded an average displacement rate in Er_2O_3 layer.

2.2. The adhesion strength evaluation of the Er_2O_3/Y_2O_3/SUS316 multi-layered MOCVD coating sample before and after Cu^{2+} ion beam irradiation at room temperature

The adhesion strength, which was one of the mechanical properties such as the soundness and durability, was evaluated using the nano-scratch testing machine (RHESCA; CSR-200). The configuration, evaluation principle and interaction of the nano-scratch testing machine were already reported in detail [12]. The relationship between the