Effect of Cu$^{2+}$ Ion Irradiation on Microstructure of Er$_2$O$_3$ Coating Layer Formed by MOCVD Method

M. Tanaka$^a$, M. Takezawa$^a$, Y. Hishinuma$^b$, T. Tanaka$^b$, T. Muroga$^b$, S. Ikeno$^c$, S.W. Lee$^c$ and K. Matsuda$^c$

$^a$Graduate School of Science and Engineering for Education, University of Toyama, Japan
$^b$National Institute for Fusion Science, Toki, Japan
$^c$Graduate School of Science and Engineering for Research, University of Toyama, Japan

Erbium oxide (Er$_2$O$_3$) coating is one of the promising methods to restrict tritium permeation and the magneto hydrodynamic pressure drop for advanced breeding blanket systems. Er$_2$O$_3$ coating layer on large interior surface area of metal pipe is deposited by using metal organic chemical vapor deposition process. In this work, the influence of Cu$^{2+}$ ion irradiation on microstructure of Er$_2$O$_3$ coating layer on Inconel 625 (SUS 316) substrate by metal organic chemical vapor deposition methods was investigated using scanning electron microscopy, transmission electron microscopy observation and X-ray diffraction analysis. Microstructure observation of Er$_2$O$_3$ coating was carried out after 0.00–1.50 dpa Cu$^{2+}$ ion irradiation at 298 K and 773 K. Scanning electron microscopy observation of the surface area on Er$_2$O$_3$ thin film revealed the crack generation on surface after Cu$^{2+}$ ion irradiation. X-ray diffraction peaks were identified in Er$_2$O$_3$ after Cu$^{2+}$ ion irradiation transmission electron microscopy observations, the formation of interlayer between Er$_2$O$_3$ coating and SUS substrate was confirmed. According to transmission electron microscopy-energy dispersive spectroscopy, the interlayer includes Fe and Cr.

1. Introduction

Nuclear fusion reactor has been focused on the new energy system [1], and the system mainly composed of superconducting coil, plasma core, and blanket system. The blanket system requires the following five conditions: (1) Not break down at high temperature. (2) Low reactivity with Li as a coolant. (3) High electrical resistivity. (4) High permeation control of tritium. (5) High electrically insulating coating of 2 µm or more. Er$_2$O$_3$ coating is one of the promising methods to restrict tritium permeation and magneto hydrodynamic (MHD) effect for advanced breeding blanket systems [2]. Hishinuma et al. reported the formation of Er$_2$O$_3$ coating layer on interior surface area of a short quartz tube using metal organic chemical vapor deposition (MOCVD) process as a new technology for broad and complicated shaped area [3]. The blanket system undergoes neutron irradiation during operation, and the Cu$^{2+}$ ion irradiation can be a reasonable candidate to simulate neutron irradiation. In this work, the Cu$^{2+}$ ion irradiation was conducted for understanding the change of insulator coating microstructure prepared via MOCVD process by neutron.

2. Experimental

Er$_2$O$_3$ coating samples on SUS 316 substrates were fabricated by the MOCVD facility in National Institute for Fusion Science (NIFS). Er$_2$O$_3$ coating was deposited with the thickness of 0.30–0.40 µm on the substrate. The Cu$^{2+}$ ion irradiation was carried out by tandem ion accelerator in Research Institute for Applied Mechanics, Kyushu University (RIAM), with 2.4 MeV of Cu$^{2+}$ ion source. The irradiation of 0.15 dpa and 1.50 dpa conducted Er$_2$O$_3$ coated samples at room temperature, and the another sample was irradiated for 0.15 dpa at 773 K. X-ray diffraction (XRD, Phillips Xpert system), scanning electron microscopes (SEM, Hitachi S3500), and transmission electron microscope (TEM, JEOL 4010T) were used for analysis of microstructure. The cross-sectional samples for TEM observation were prepared by focus ion beam (FIB, Hitachi FB-2100) method.

3. Results and discussion

3.1. XRD analysis

Figure 1 shows the results of XRD analysis for four samples. All peaks have a good agreement with Er$_2$O$_3$ and SUS 316 peaks after each irradiative conditions. These profiles reveal that Er$_2$O$_3$ coating layer has a good neutron irradiation stability.

![Fig. 1. XRD analysis of four Er$_2$O$_3$ samples: (a) 0.00 dpa–298 K, (b) 0.15 dpa–298 K, (c) 1.50 dpa–298 K and (d) 0.15 dpa–773 K.](image_url)
3.2. Microstructure observation: Cu$^{2+}$ ion irradiation: 0.00 dpa and 0.15 dpa at 298 K

Figure 2a shows the deposited Er$_2$O$_3$ particles from the surface of sample with SEM. With irradiation of 0.15 dpa at room temperature, surface morphology is barely changed in Fig. 2c. From cross-sectional images of TEM, the dendritic structure was observed in the Er$_2$O$_3$ layer and coating thickness is about 0.40 µm in Fig. 2b.

3.3. Microstructure observation: Cu$^{2+}$ ion irradiation: 1.50 dpa at 298 K

Figure 3a shows drastic changing of surface morphology after Cu$^{2+}$ ion irradiation of 1.50 dpa. After 1.50 dpa of irradiation, it is apparent that crack generation occurred during Cu$^{2+}$ ion irradiation. With increase of the degree of the irradiation from 0.15 dpa to 1.50 dpa, the thickness of Er$_2$O$_3$ coating decreased to 0.30 µm in Fig. 3b and surface structure changed remarkably. It was possible to observe the decrease of white layer between Er$_2$O$_3$ layer and SUS 316 substrate from cross-sectional image after 1.50 dpa of Cu$^{2+}$ ion irradiation. According to TEM-EDS profiles in Fig. 3c, the layer includes Fe and Cr. Also, 20–30 at.% Cu element was found from Er$_2$O$_3$ layer and was decreased as going down to SUS 316 substrate. Cu elements was found from both Er$_2$O$_3$ and SUS 316. It means that some Cu$^{2+}$ ion can permeate throughout Er$_2$O$_3$ coating.

3.4. Microstructure observation: Cu$^{2+}$ ion irradiation: 0.15 dpa–773 K

In Fig. 4a, no great change of surface morphology was observed after 0.15 dpa of Cu$^{2+}$ ion irradiation at 773 K. After close inspection of TEM-EDS profiles in Fig. 4c, the unknown layer includes Fe and Cr. Also, 10–20 at.% Cu element was found in Er$_2$O$_3$ layer and was decreasing as going down to SUS 316 substrate, the same as in Fig. 3c.

4. Conclusions
1. Er$_2$O$_3$ coating has good stability for neutron irradiation from XRD analysis.
2. After 1.50 dpa neutron irradiation, it was confirmed that surface structure remarkably changed.
3. After close inspection of TEM-EDS, unknown layer includes Fe, Cr and Cu element throughout both Er$_2$O$_3$ layer and SUS 316 substrate.

References