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Effect of Cu^{2+} Ion Irradiation on Microstructure of Er_2O_3 Coating Layer Formed by MOCVD Method

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Erbium oxide (Er_2O_3) coating is one of the promising methods to restrict tritium permeation and the magneto hydrodynamic pressure drop for advanced breeding blanket systems. Er_2O_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_2O_3 coating layer on stainless steel 316 (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_2O_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_2O_3 thin film revealed the crack generation on surface after Cu^{2+} ion irradiation. X-ray diffraction peaks were identified in Er_2O_3 after Cu^{2+} ion irradiation transmission electron microscopy observations, the formation of interlayer between Er_2O_3 coating and SUS substrate was confirmed. According to transmission electron microscopy-energy dispersive spectroscopy, the interlayer includes Fe and Cr.

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PACS/topics: nuclear fusion, coating, Er2O3, thin film, TEM, SEM and XRD

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_2O_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_2O_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_2O_3 coating samples on SUS 316 substrates were fabricated by the MOCVD facility in National Institute for Fusion Science (NIFS). Er_2O_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_2O_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_2O_3 and SUS 316 peaks after each irradiative conditions. These profiles reveal that Er_2O_3 coating layer has a good neutron irradiation stability.

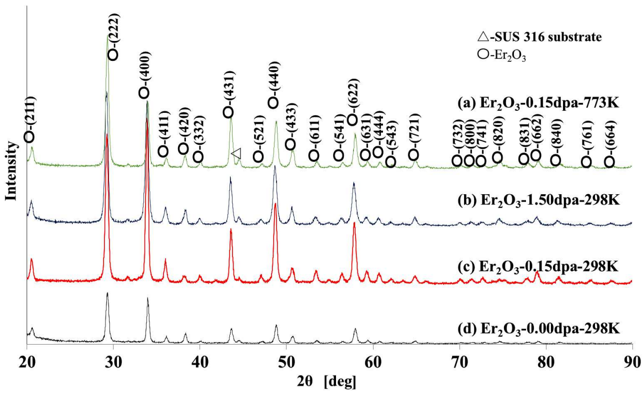

Fig. 1. XRD analysis of four Er_2O_3 samples: (a) 0.00 dpa–298 K, (b) 0.15 dpa–298 K, (c) 1.50 dpa–298 K and (d) 0.15 dpa–773 K.

3.2. *Microstructure observation: Cu^{2+} ion irradiation: 0.00 dpa and 0.15 dpa at 298 K*

Figure 2a shows the deposited Er_2O_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_2O_3 layer and coating thickness is about 0.40 μm in Fig. 2b.

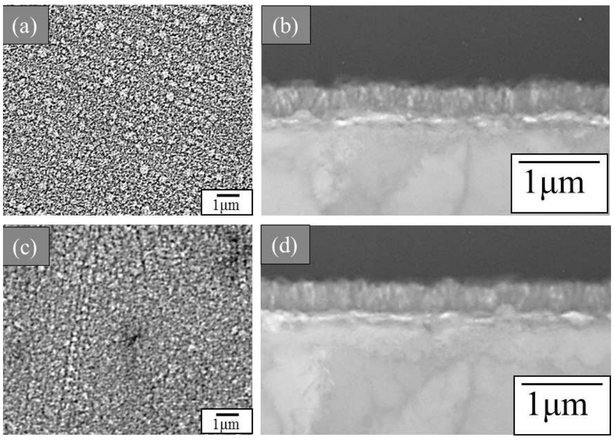

Fig. 2. Surface morphology and microstructure observations for Er_2O_3 films: (a),(b) 0.00 dpa–298 K and (c),(d) 0.15 dpa–298 K. (a),(c) and (b),(d) are SEM, TEM images, respectively.

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_2O_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_2O_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_2O_3 layer and was decreased as going down to SUS 316 substrate. Cu elements was found from both Er_2O_3 and SUS 316. It means that some Cu^{2+} ion can permeate throughout Er_2O_3 coating.

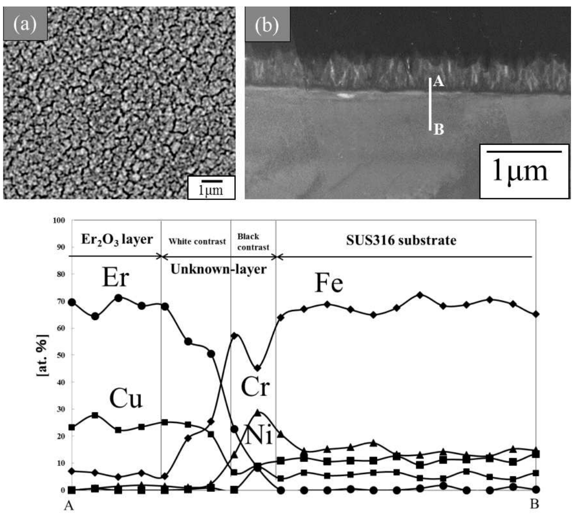

Fig. 3. (a) SEM image of surface morphology, (b) TEM image of cross-section for Er_2O_3–1.50 dpa at 298 K and (c) TEM-EDS profiles for Er, Fe, Cr, Ni, and Cu elements obtained for line A–B in (b).

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_2O_3 layer and was decreasing as going down to SUS 316 substrate, the same as in Fig. 3c.

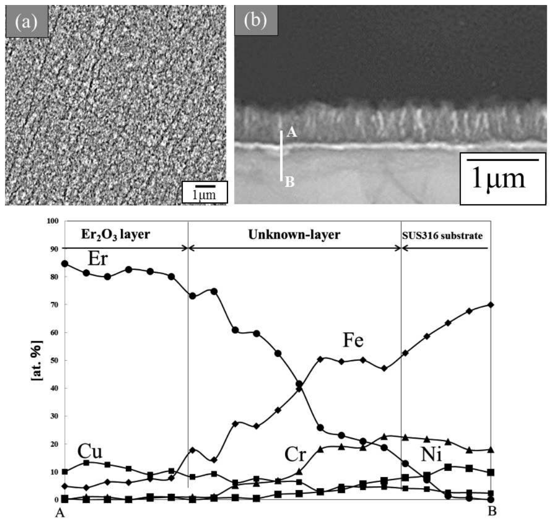

Fig. 4. (a) SEM image of surface morphology and (b) TEM image of cross-section for Er_2O_3–0.15 dpa at 773 K and (c) TEM-EDS profiles for Er, Fe, Cr, Ni, and Cu elements obtained for line A–B in (b).

4. Conclusions

1. Er_2O_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_2O_3 layer and SUS 316 substrate.

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