§3. Formation and Nanostructure of MOCVD Processed Er<sub>2</sub>O<sub>3</sub>/oxide Buffer Double Stacked Insulator Coating

Hishinuma, Y., Tanaka, T., Muroga, T., Sagara, A., Shinkawa, T., Matsuda, K. (Univ. Toyama)

Some ceramic materials such as CaO,  $Y_2O_3$ , CaZrO<sub>3</sub>, AlN and  $Er_2O_3$  have been studied as candidates for the insulating coating of liquid metal (Li and Pb-Li) breeding blanket system<sup>1</sup>). In order to develop coating methods of  $Er_2O_3$  layer on a large area and complex shape (for example: inner surface of the complex-shaped duct tubing) for the advanced liquid metal breeder blanket application, the metal organic chemical vapor deposition (MOCVD) process was investigated as the new technology of coating for possible application to blankets<sup>2</sup>). We successfully formed a homogeneous  $Er_2O_3$  thin coating layer on Si single crystal, vanadium and austenitic stainless steel 316 (SUS316) substrates through the MOCVD process<sup>2</sup>). However, it is difficult to form a thick coating on metal substrate with epitaxial growth, like in chemical vapor deposition (CVD).

We approached and investigated to form a double stacked coating layer on the SUS316 substrate using an intermediate oxide layer (buffer layer) for the formation of a thicker  $Er_2O_3$  coating with high texture orientation. The both yttrium oxide (Y<sub>2</sub>O<sub>3</sub>) and cerium oxide (CeO<sub>2</sub>), having similar lattice constants to those of  $Er_2O_3$  crystal and SUS substrate materials, were selected as the buffer layer of double stacked coating. In this report, the effect of CeO<sub>2</sub> buffer layer formation on the nanostructure of double stacked coating layer ( $Er_2O_3/CeO_2/SUS$  substrate) was investigated.

After the  $Er_2O_3$  MOCVD deposition on the CeO<sub>2</sub>/SUS substrate, Transmission Electron Microscope (TEM) observations were performed with the TOPCON EM-002B electron microscopes operated at an accelerating voltage of 200 kV. The constituent element spatial distributions were

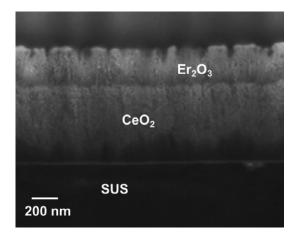


Fig. 1 TEM bright field images of cross-sectional area on  $Er_2O_3/CeO_2$  double stacked coating.

analyzed using a combination of TEM and energy dispersive X-ray spectroscopy (TEM-EDS). TEM samples of the double stacked layer were prepared using a focused ion beam (FIB) mill. The FIB milling was performed with HITACHI FB-2000K operated at an accelerating voltage of 30 kV. Observation planes were perpendicular (crosssection) to the surface of the double stacked layer.

TEM bright field images of cross-sectional area on Er<sub>2</sub>O<sub>3</sub>/CeO<sub>2</sub> double stacked coating are shown in fig. 1. We confirmed clearly that Er<sub>2</sub>O<sub>3</sub> was formed on CeO<sub>2</sub> buffer. However, the thicknesses of Er<sub>2</sub>O<sub>3</sub> layers on CeO<sub>2</sub> was about 300 nm, and this thickness was decreased compared with non-buffer sample. Fig. 2 shows high magnification TEM bright field images and electron diffraction patterns of cross-sectional area of Er<sub>2</sub>O<sub>3</sub>/CeO<sub>2</sub> double stacked layer. The texture of CeO<sub>2</sub> buffer layer showed a "granular"-like texture. We found that the "granular"-like CeO<sub>2</sub> crystals were grown to [111] direction. The "columnar"-like Er<sub>2</sub>O<sub>3</sub> crystals were grown on the surface of CeO<sub>2</sub> buffer layer. Er<sub>2</sub>O<sub>3</sub> grain growth direction was also obtained to be [110]. These suggested that the "columnar"-like Er<sub>2</sub>O<sub>3</sub> crystal was grown on the "granular"-like CeO<sub>2</sub> crystals and the growth direction of Er<sub>2</sub>O<sub>3</sub> crystal corresponded to the CeO<sub>2</sub> crystals.

Smith, D.L. et al.: J. Nucl. Mater. **307–311** (2002) 1314.
Hishinuma, Y. et al.: J. Nucl. Mater. **417** (2011) 1214.

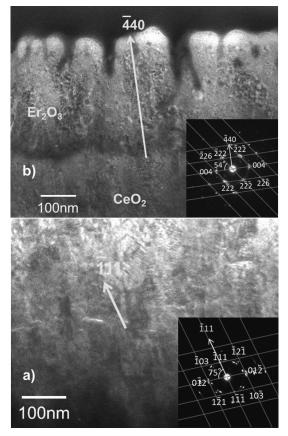


Fig. 2 TEM bright field images and electron diffraction patterns of cross-sectional area on  $Er_2O_3/CeO_2$  double stacked coating. ((a) center of  $CeO_2$  buffer layer and (b) interface between  $Er_2O_3$  and  $CeO_2$  buffer layers.