§7. Adhesion Strength of Er₂O₃/buffer stacked MOCVD Ceramic Coating for advanced Breeding Blanket

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The electrical insulator and tritium permeation barrier coatings are one of the critical materials for advanced blanket systems. Erbium oxide (Er_2O_3) is well known as the one of the candidate oxide coating materials due to the higher electrical resistivity at higher temperature and significant suppression of the tritium leakage. We have therefore been developed for Er_2O_3 layer coating process on broad and complicated shaped components, which were blanket structures and duct pipes. Recently, we succeeded to establish Er_2O_3 coating formation into the stainless-steel pipe interiors using metal organic vapor deposition (MOCVD) process.

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 important issue to investigate the blanket design and fabrication process. We approached to evaluate the adhesion strength, which was one of the important mechanical properties on the coating materials, in order to estimate of soundness and durability as the blanket component material.

Generally, the cross-cut adhesion and the pull off methods are not applied to evaluate the adhesion strength of the hard coating materials like the ceramic. 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. Furthermore, the adhesion strength evaluation of the several hard coatings by the scratch method was qualified to the Japan Industrial Standard (JIS) R3255. We evaluated the adhesion strength of the MOCVD processed Er_2O_3 /buffer multi-stacked coating on the stainless steel (SUS316) substrate using nano-scratch method.

In order to investigate the soundness of coating layer, the adhesion strength, which was one of the soundness and durability factors, was evaluated using the nano-scratch tester (RHESCA; CSR-200). On the nano-scratch mechanism, the relationship between the dynamic load (W) applied to the coating and the shearing stress (F_s) on the interface the coating layer and substrate have established as the formula of Benjamin and Weaver following as (1)

$$F_s = H_b / \sqrt{(\pi R^2 H_b / W) - 1}$$
 ----- (1)

where *R* is the curvature radius of the diamond stylus, and H_b indicates Brinell hardness of the substrate. As the dynamic load to the coating (*W*) is increased, the shearing stress (*F_s*) on the boundary between coating and substrate increases. This minimum dynamic load when the coating

layer breaks and/or separates from the substrate was defined as the critical adhesion strength (W_c) in this study.

Fig. 1 shows that typical scratch trace image and scratch test data of Er₂O₃/Y₂O₃ buffer multi-stacked coating using the nano-scratch tester. (a) The CCD image of the nano-scratch trace on the Er₂O₃/Y₂O₃ multi-stacked coating layer and (b) the displacement diagram between sensor output and dynamic load force. We found that the scratch trace corresponded with the displacement diagram between the sensor output and the applied scratch force. The excitation amplitude of the stylus became smaller with the increase in the applied scratch force. At the same time, the sensor output was also increased with the decrease in the excitation amplitude. Here, the sensor output indicates friction force between the stylus and coating surface. The raw sensor output data was converted using fast Fourier transformation. On the other hands, the sensor output was increased with increasing applied scratch force and the two significant increases of sensor output coincided with the two separation points of the scratch trace. The first separation point indicated the adhesion between Er₂O₃ and Y₂O₃, and the second separation point also indicated the adhesion between Y2O3 and SUS316 substrate. We found that the origin and strength of fracture and adhesion between each coating layer could be separated by the adhesion strength (W_c) . In this way, nano-layer scratch instrument is available to evaluate the adhesion strength of the multi-stacked thin coating.



Fig.1 Typical scratch trace image and scratch test data of Er_2O_3/Y_2O_3 buffer multi-stacked coating using the nano-scratch tester.