§4. Establishment of the Adhesion Strength Evaluation of Oxide Insulator Coating Using Nano-scratch Method

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1. Introduction and Motivation

The electrical insulator and hydrogen permeation barrier coatings are important materials to realize the liquid metal and molten-salt typed breeding blanket systems. We found that erbium oxide (Er_2O_3) is one of the promising materials as the electrical insulator and hydrogen permeation restraint coatings. Recently, we succeeded to form homogeneous Er_2O_3 coating using Metal Organic Chemical vapor Deposition (MOCVD) process as the new technology for Er_2O_3 coating on large broad and complicated shaped components including duct interiors. Establishing the mechanical property evaluation method for insulator coating is extremely important to certify the durability of coating material in the blanket systems. The adhesion strength property, which is one of the key mechanical and durability properties of coating materials, was investigated using the nano-scratch method.

2. The evaluation principle of the nano-scratch method

In order to evaluate adhesion strength of the MOCVD processed Er_2O_3 coating samples, nano-scratch tests were carried out using nanolayer scratch instrument (CSR-2000: RHESCA Co.,Ltd). The schematic image of the scratch mechanism of nano-scratch instrument is shown in Fig. 1. In these tests, the surface of the coating layer is scratched by a vibrating diamond stylus tip of a given curvature radius mounted on an elastic arm while the stylus is being lowered in the coating thickness direction. The elastic arm is deformed because of the lowering movement of the stylus, resulting in an increased load force to the coating layer applied by the stylus. According to the formula of Benjamin and Weaver, the relationship between the dynamic load (W) applied by the stylus to the coating and the shearing stress (F_s) on the interface the coating layer and substrate have established as the following Eq. (1) [1],

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

where R is the curvature radius of the 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.

When the shearing stress exceeds the adhesive strength of the coating layer, the coating layer on the substrate is broken and/or separated from the substrate. This minimum dynamic load when the coating layer breaks and/or separates from the substrate is defined as the critical adhesion force (W_c).

Typical scratch trace image and test data of Er_2O_3 coating layer using nano-layer scratch instrument are shown in Figs. 2 (a) and (b). The scratch trace image was taken by CCD camera attached to the nano-layer scratch instrument. We found that the scratch trace corresponded with the displacement diagram between the sensor output and 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. On the other hands, the sensor output was increased with increasing applied scratch force and the significant increase of sensor output coincided with the separation point of the scratch trace. We defined the applied force at the significant increase of sensor output as the W_c. We found that nano-layer scratch instrument is available to evaluate the adhesion strength of the Er_2O_3 thin coating.

[1] P. Benjamin and C. Weaver, Proc. R. Soc. London, Ser. A **254**, 1227, (1960), 163.

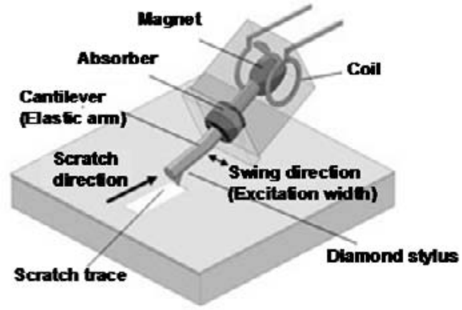

Fig.1 Typical schematic image of the scratch mechanism of nano-scratch instrument (CSR-2000, RHESCA Co.,Ltd)

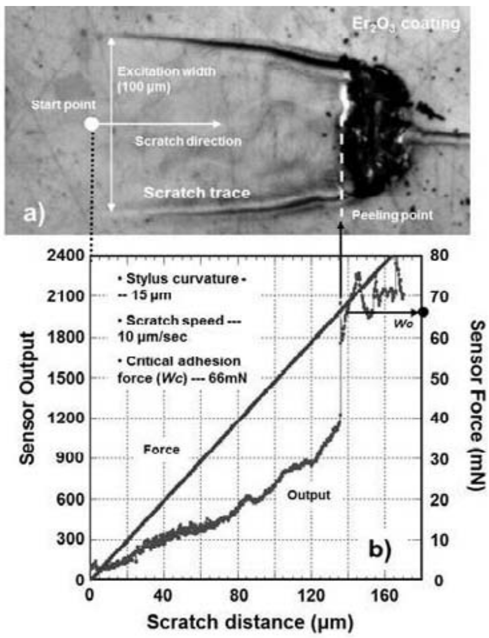

Fig.2 Typical scratch trace image and test data of Er_2O_3 coating layer using the nano-layer scratch instrument. (a) The CCD image of the nano-scratch trace and (b) the displacement diagram between sensor output and force.