

§1. Improvement of a Fabrication Process of MOD Ceramic Coatings on a Low Activation Ferritic Steel

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For suppression of MHD pressure drop and hydrogen isotope permeation in liquid self-cooled blanket systems, development of large area ceramic coatings is being conducted. Among various coating techniques, the metal organic decomposition (MOD) method using an organic liquid is considered suitable for the flexibility in a coating fabrication process on large and complicated blanket components. One of issues in the coating development with the MOD method is control of substrate oxidation. A baking process at $>450^\circ\text{C}$ in an oxidizing atmosphere is necessary for removal of organic components of an MOD liquid and production of an oxide ceramic layer. However, this process also oxidize a metal substrate surface. In our previous studies on coating fabrication on low activation ferritic steel (JLF-1: Fe-9Cr-2W) substrates, a Fe_2O_3 or Cr_2O_3 oxidation layer was produced on a substrate surface, i.e. under a ceramic coating layer, depending on the temperature and oxygen partial pressure in the baking process.¹⁾ It has been confirmed that the production of a Fe_2O_3 layer under an oxide ceramic layer degrades the coating performance significantly.²⁾ In the fiscal year of 2013, process improvement in MOD Er_2O_3 coating fabrication on a substrate of the low activation ferritic steel JLF-1 has been studied.³⁾

A JLF-1 substrate was heated up to 700°C in air at the pressure of $<5 \times 10^{-3}$ Pa and kept for 60 minutes. Then the pressures was increased to 5 Pa and kept for 60 minutes. After the heat treatment, a Cr_2O_3 of $0.2\ \mu\text{m}$ in thickness was obtained on the JLF-1 substrate surface. The Cr_2O_3 layer was significantly stable and almost no change was observed even after a heat treatment at 700°C in air. In the heat treatment in air, a Fe_2O_3 layer was produced on a bare JLF-1 substrate. Therefore the Cr_2O_3 surface layer is considered to be a barrier to prevent further production of a Fe_2O_3 layer in heat treatments at high temperatures in air.

An Er_2O_3 layer was fabricated on the Cr_2O_3 covered JLF-1 substrate by a dip coating technique (Fig. 1). The substrate was dipped into an MOD liquid and withdrawn at the speed of 100mm/min . Then the substrate was dried at 120°C and baked at 475°C in air. The dipping, drying and baking processes were repeated 20 times in air. In our previous studies, the baking process had to be performed in $\text{Ar}+10\text{ppm O}_2$ atmosphere at 700°C to prevent the production of Fe_2O_3 layer on the substrate surface.²⁾ In the present study, all the coating processes could be performed in air, since a JLF-1 substrate was covered with a stable Cr_2O_3 barrier-layer in the pretreatment described above and prevents further oxidation during the coating baking process. The thickness of the obtained Er_2O_3 layer was $0.75\ \mu\text{m}$.

Hydrogen permeation tests²⁾ have been performed for the Cr_2O_3 covered JLF-1 substrate (Sample #1: JLF-

$1+\text{Cr}_2\text{O}_3$) and that with an MOD Er_2O_3 coating (Sample #2: JLF-1+ Cr_2O_3 + Er_2O_3). Results of the permeation tests are shown in Fig. 2. The hydrogen permeability through Sample #1 was almost 2 orders lower than that of bare JLF-1 plate. This indicates that the Cr_2O_3 layer on a JLF-1 substrates has a function as a permeation barrier. The magnitude of suppression by Sample #2 was similar to that of Sample #1 at lower than 550°C . However, the permeability decreased slightly at 600°C . Since the MOD Er_2O_3 coating was baked at 475°C , it is considered that the crystallinity of the Er_2O_3 layer was improved during the measurement at 600°C and the performance as the permeation barrier was also improved.

The heat treatment to produce the Cr_2O_3 layer on a JLF-1 substrate surface improved the flexibility of the following coating process and shortened the fabrication time. Considering the heat treatment in the fabrication process of JLF-1, the baking of MOD coatings up to 700°C in air would be allowable. This baking at high temperatures will improve the crystallinities of the MOD Er_2O_3 coatings and superior permeation barrier performances could be obtained in the future study.

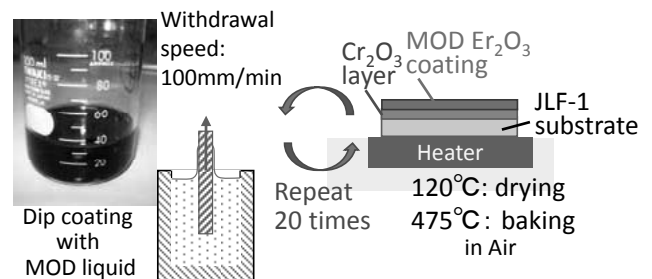


Fig. 1 Fabrication of an MOD Er_2O_3 coating on a Cr_2O_3 covered JLF-1 substrate.

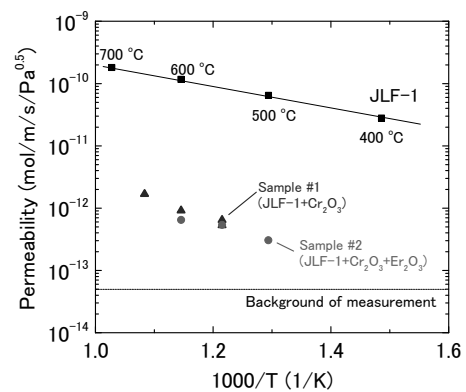


Fig. 2 Comparison of hydrogen permeabilities through a bare JLF-1 plate, Sample #1 (JLF-1+ Cr_2O_3) and Sample #2 (JLF-1+ Cr_2O_3 + Er_2O_3).

1) T. Muroga et al., Fusion Science and Technology 64 (2013) 211-215.

2) D. Zhang et al., Fus. Sci. Tech. 60 (2011) 1576-1579.

3) T. Tanaka et al., presented at 16th International Conference on Fusion Reactor Materials (ICFRM16), 20-26 October, 2013, Beijing. Submitted to Journal of Nuclear Materials.