# §27. Investigation on the Optimization in the Hydrogen Permeation Measurement Technique of the Oxide Coatings

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Yttrium oxide ($Y_2O_3$) is one of the candidate of materials for tritium permeation barrier (TPB) due to its high permeation reduction factor and lower radioactivation characteristics than erbium oxide ($Er_2O_3$). In this study, we performed fabrication, characterization and deuterium permeation experiments for $Y_2O_3$ coatings.

$Y_2O_3$ coatings were fabricated on reduced activation ferritic/martensitic steel F82H plates by metal organic decomposition (MOD) with an yttrium oxide coating precursor (Kojundo Chemical Laboratory Co., Ltd. Y-03Ⓡ). The MOD procedure established in Ref. [1] was shown in Fig. 1. Cross section of the coatings was observed by scanning electron microscope (SEM), and crystal structure of the coatings was analyzed by X-ray diffraction (XRD).

Deuterium permeation experiments were performed with a gas-driven permeation apparatus at 300–700 ºC. The permeation flux per unit area at steady-state $J$ (mol $m^{-2}$ $s^{-1}$) through a sample with a thickness of $d$ (m) is expressed by following equation:

$$J = P\,(p^{0.5} / d) \qquad (1)$$

$P$ (mol $m^{-1}$ $s^{-1}$ $Pa^{-0.5}$) is the permeability, and $p$ (Pa) is the driving pressure. An exponent of driving pressure is estimated by fitting the permeation fluxes at different driving pressures. For evaluation of TPB efficiency, a permeation reduction factor is calculated dividing the permeation flux of a bare substrate by that of a coated one.

In XRD spectra of the coated samples shown in Fig. 2, peaks derived from yttrium oxide were observed in the coating heat-treated at 700 ºC, but not observed in the coating heat-treated at 670 ºC. Therefore, the microstructure of the coatings varied with heat-treatment temperature: amorphous at 670 ºC and crystallized at 700 ºC. Deuterium permeation flux of the amorphous coating was an order of magnitude lower than the uncoated steel at 500 ºC, while that of the crystallized coating was two orders of magnitude lower at 300–550 ºC. From these results, MOD coatings especially amorphous coating did not secure sufficient permeation reduction factor in the low temperature region. However, the permeation fluxes of both coatings were drastically decreased during the measurements at higher temperatures (600-700 ºC) by a factor of up to 1000, although the thickness of the coatings was only approximately 250 nm. These factors are considered sufficient for an actual use as TPB. In addition, a microstructure modification occurred during the permeation measurement under a high-temperature condition with hydrogen flux. Temperature dependence of deuterium diffusivity in the coatings suggests that the decrease of the permeation flux has been derived from a decrease of the diffusivity. Characteristic permeation behaviors were observed with different heat-treatment conditions; however, they can be interpreted using the permeation mechanism clarified in the previous $Er_2O_3$ coating studies.

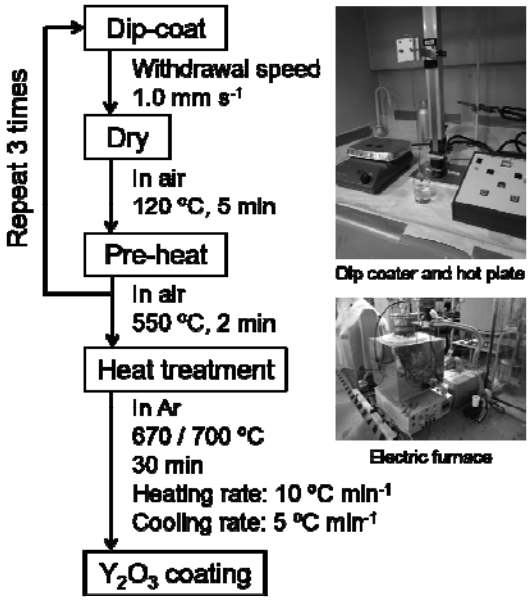


Fig. 1. Metal organic decomposition process.

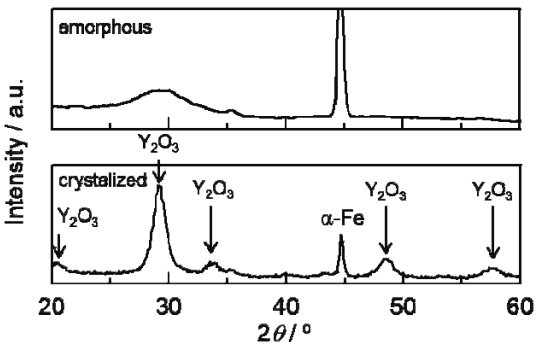


Fig. 2. XRD spectra of amorphous and crystalized $Y_2O_3$ coatings.

1) Chikada, T. et al.: Journal of Nuclear Materials **442** (2013) 533.