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Study of hydrogen absorption and desorption properties of oxygen-free Pd/Ti thin film as a non-evaporable getter (NEG) coating by using nuclear reaction analysis (NRA)

Absorption and desorption properties of hydrogen for the oxygen-free Pd/Ti thin film was studies by using $^{1}H(^{15}N,\alpha\gamma)^{12}C$ nuclear reaction analysis(NRA) and thermal desorption spectroscopy (TDS).
The oxygen-free Pd/Ti film was developed as a new non-evaporable getter (NEG) coating, which is expected to keep high pumping speed after many cycles of air-vent and activation by heating [1].
It is expected that non-activated dissociation of H_2 on the Pd surface and diffusion into Ti film can occur at room temperature.
It is also expected that hydrogen can be desorbed from Ti film through Pd at lower temperature than the conventional NEG.
Since TiO_2 works as a hydrogen barrier, it is important that Pd/Ti thin film is produced under oxygen free condition, which is realized by sublimation of Ti and Pd on stainless steel (SS304) in ultra-high vacuum (UHV) . Pumpdown curves of the Pd/Ti film coated SS304 was measured and the pumping speed was confirmed to be almost constant under several venting-pumpdown-baking cycles. This coating is expected to overcome a weakness of the conventional NEG, whose pumping speed is degraded rapidly by repeated heating-venting cycles. Since H_2 is main residual gas under UHV condition, it is important to measure hydrogen absorption/desorption properties of the Pd/Ti thin film precisely [1].

The NRA using $^{1}H(^{15}N,\alpha\gamma)^{12}C$ is suitable for investigating depth profile of hydrogen concentration quantitatively. The narrow energy resonance at 6.385 MeV of nuclear reaction of ^{15}N with hydrogen is deteriorated by Doppler-broadening and struggling effect, but high resolution (a few nm) depth profiling of H can be achieved near the surface. The Pd(6 nm)/Ti(12 nm)/SS304L (8 mm × 8 mm × 1 mm) sample was prepared in a UHV chamber, in which film thickness was estimated by transmission electron microscopy (TEM). The energy dispersion X-ray spectroscopy mapping using TEM (TEM-EDX) shows that the layers of Pd and Ti are separated. The NRA spectrum obtained just after evacuation revealed that the small amount of H exists at the Pd surface and in the Ti thin film. H in the Ti film increased 20 times by exposing to 10000 L H_2. A part of H_2 may be dissociated at the W filament of the vacuum gauge. By the quantitative analysis, it is suggested that $TiH_{1.27}$ is produced in the whole Ti layer. Though the density of H in the Ti film did not change by heating below 200 °C, it rapidly decreased above 200 °C. Repeated cycles of absorption at room temperature and desorption by annealing at about 240 °C was possible. However, H absorption ability was lost, once the sample was annealed above 500 °C.

[1] T. Miyazawa, K. Tobishima, H. Kato, M. Kurihara, S. Ohno, T. Kikuchi and K. Mase, Vac. Surf. Sci. 61, 227 (2018).

Primary author(s) : Dr MATSUMOTO, Masuaki (Tokyo Gakugei University)

Co-author(s) : Mr OKADA, Tomohiro (Tokyo Gakugei University); Mr MIYAZAWA, Tetsuya (SOKENDAI); Prof. MASE, Kazuhiko (KEK); Dr HASHIMOTO, Ayako (National Institute for Materials Science); Prof. WILDE, Markus (Institute of Industrial Science, University of Tokyo); Prof. FUKUTANI, Katsuyuki (Institute of Industrial Science, University of Tokyo)

Presenter(s) : Prof. FUKUTANI, Katsuyuki (Institute of Industrial Science, University of Tokyo)

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