§6. Study on Formation of Metal-carbon Mixed Deposition Layer and Hydrogen Isotope Behavior

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Evaluation of hydrogen retention in deposition layers formed in a plasma vessel is an important issue from viewpoints of fuel control and radiation safety. Many studies about hydrogen retention in carbon deposition layers have been performed. However, hydrogen retention in metal deposition layer and metal-carbon mixed layer have not been clarified yet. In this study, deposition layers were formed from type 316 stainless steel (SS) by a sputtering method using hydrogen-argon plasma or methane-hydrogen plasma. Hydrogen retention in the deposition layer was investigated by a thermal desorption method in argon atmosphere.

Fig.1 shows a schematic diagram of the experimental apparatus constructed for formation of a deposition layer. A cylindrical quartz tube, 400 mm in length and 10 mm in inner diameter, was used as a plasma reaction tube. The tube has side-arm to insert a SS target connected to a DC power supply. A quartz substrate, 5×10 mm^2 in size and 1 mm in thickness was set near the SS target. The tube was evacuated by a rotary pump and then a feed gas was introduced via a mass flow controller. Impurity water vapor in a gas cylinder was excluded by Molecular Sieve 3A. The pressure in the tube was adjusted by varying the conductance of evacuation using a leak valve. Plasma was generated by an inductive coupling method.

In preliminary test, the SS target was exposed to hydrogen plasma for a several days. However, an enough amount of deposition was not formed because the sputtering yield was too small. For this reason, 2 % H_2 with Ar gas or 11% CH_4 with H_2 was used as a feed gas. The deposition rate was effectively increased by using these mixed gases. When the substrate was exposed to plasma, the temperature of substrate increased to about 100 ^oC. In order to investigate the temperature dependence of hydrogen retention, the external heating was conducted. When a metal-carbon mixed deposition layer was produced, the SS target was inserted from the downstream side as shown in Fig.1.

The substrate on which the deposition layer was formed was placed into a quartz tube connected to a gas chromatograph (GC). The tube was filled with Ar gas and then heated to 800 ^oC in 100 ^oC step. Every 20 minutes, the tube was purged with Ar. Gaseous components in the purge gas were measured by the GC.

Elemental analysis in deposition layers was carried out by EDX. The ratio of metal atoms (Fe, Ni, Co, Mo) was almost same as the ratio in 316 stainless steel. The metal-carbon mixed layer (M-C layer) contained carbon of 18 wt %. The release behavior of hydrogen from M-C layer is shown in Fig.2. The release begun from 200 ^oC and it continued 800 ^oC. Total amount of released hydrogen was $\times 10^{-6}$ mol. Atomic ratio, H/M was estimated to be 0.12. The release behavior of hydrogen from metal layers formed by hydrogen-argon plasma was approximately same as that from M-C layer. The values of H/M in the deposition layers formed at different temperatures are compared in Fig.3. Literature data in this figure has been obtained by the parallel plate sputtering devise[1]. In this experiment, an effect of carbon was not observed. The carbon dependence on hydrogen retention will be investigated in near future.

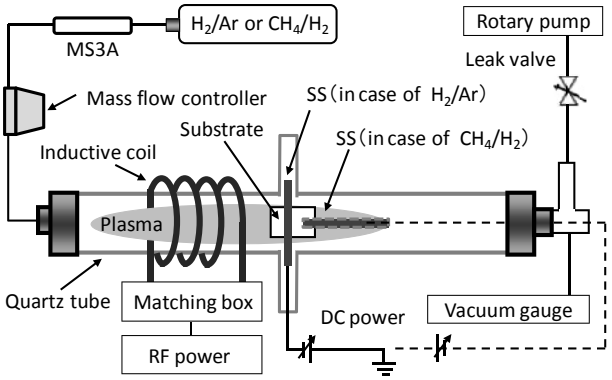

Fig.1 Schematic diagram of the experimental apparatus.

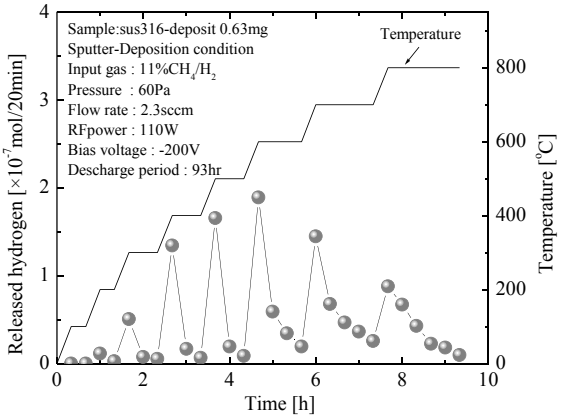

Fig.2 Hydrogen release from metal-carbon mixed layer.

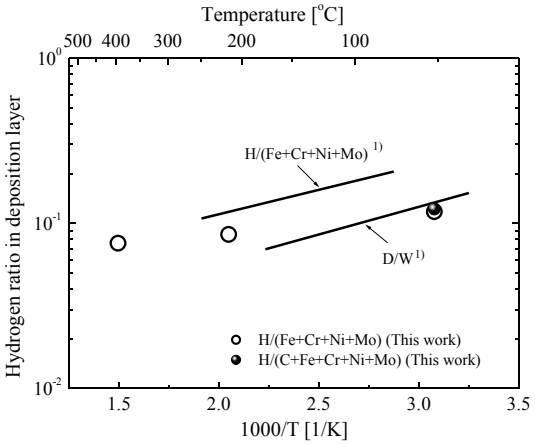

Fig.3 Hydrogen retention in deposition layers.

1) K. Katayama, Y. Uchida, T. Fujiki, M. Nishikawa, S. Fukada, N. Ashikawa, T. Uda, J. Nucl. Mater., 390-391 (2009) 689-692.