Observation of the effect of deuterium charge in the modeled metal bilayers of Fe/TiN using the neutron reflectometer MINE

M. Yamada[A], T. Nakayama[B], M. Hino[C], K. Ikeda[D], K. Mori[A]

[A]IMSS, KEK, [B]Formerly: Kobe Steel, Ltd., [C]KURNS, Kyoto Univ., [D]CROSS

INTRODUCTION:

As technology for light-weighting structural metal materials and hydrogen infrastructure advances for global environmental protection, addressing hydrogen embrittlement in high-strength structural materials is an urgent issue. Understanding of the mechanism of hydrogen embrittlement and its prevention is essential for developing high-performance steels that combines high strength and resistance to hydrogen embrittlement.

This study aims to elucidate the behavior of hydrogen trapping by using a metallic multilayer film that models the interface between the nanoprecipitates and the matrix. One kind of hydrogen embrittlement-resistant alloys has a system in which nano-precipitates (such as TiN and TiC) are dispersed in steel matrix [1,2].

Experimental setup:

The metallic multilayer samples were deposited by using ion beam sputtering at KURNS [3]. The sample consisted of 40 repetitions of the bilayer of Fe (t0.5 nm) and Ti (t0.5 nm) on glass or Si substrate. Because the combination of multilayers with Fe and TiN did not show significant changes by hydrogen(H)/deuterium(D) charge in the preceding study, we modified the material of TiN layer to Ti because one of hydrogen storage materials is FeTi(H). The Ti-layers were sputtered under hydrogen-mixed atmosphere. The sample area was 40x50 mm^2.

Deuterium was charged to the multilayers by cathodic electrolysis at a current density of 2 mA/cm^2 for 30 minutes using a deuterium aqueous solution of 3mass% NaCl + 0.01mol/l KSCN.

X-ray Reflectivity result:

Prior to the neutron reflectivity measurement (NR) at the MINE beamline, X-ray reflectivity (XR) was used to determine the thicknesses of multi-layers. The results showed that the total thickness of the Fe/Ti(H) bilayer increase from 1.18 nm to 1.23 nm after the D charge, corresponding to 4% increase of total thickness.

Neutron Reflectivity (NR) Results:

Finally, we conducted NR measurement at C3-1-2 (MINE), JRR-3 to observe changes of material composition after D charge by observing the critical angle that defines the Scattering Length Density (SLDs) of layers. The NR result showed the significant increase in the critical angle by 5% between before and after the D charge (See Fig. 1). This result suggests that substantial amount of trapped D exist in the multilayers after charging, as D has positive SLD.

Conclusion:

The XR and NR results confirmed that significant amount of D was successfully charged into the [Fe/Ti(H)] multilayers, and that XR and NR are effective tools for this analysis. The next step involves distinguishing the difference in SLD between D and H charge using NR. Further studies will optimize the model multilayers to precisely identify the location and status of charged D or H.

Reference:

[1] Takenori Nakayama et al., Materia, **41** (3) p.230 (2002)

[2] Japanese Patent No.2932943, etc.

[3] M. Hino, et al., Nucl. Instr. and Meth., **797**, 265 (2015).

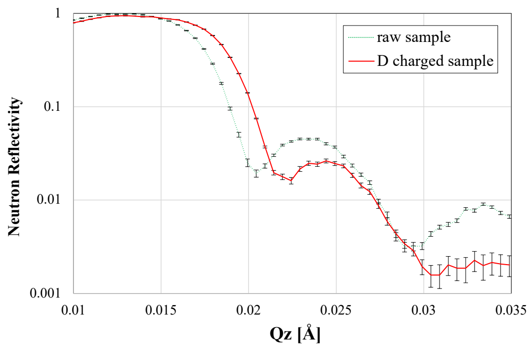

Fig. 1. Neutron Reflectivity profiles of the [Fe/Ti]x40 multilayers before and after the D charge.