# §33. Deuterium Retention in Tungsten Nitride under Low-Energy Deuterium-plasma Exposure

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Formation of tungsten nitride layers on W wall and oxygen inclusion is anticipated under N-plasma discharge in order to remove hydrogen isotopes. Thus, deuterium (D) retention in $WNO_x$ is important for designing the devices. We have measured deuterium retention in $WNO_{.4}$ films.

Samples of $WNO_{.4}$ films were prepared on the c-plane-cut $Al_2O_3$ substrate at the substrate temperature of 425ºC using a RF-sputter deposition method with W target and $N_2$ gas. X-ray diffraction (XRD) shows that the films have hexagonal WN structure. XRD peaks corresponding to $WO_3$ and W were not observed. W density of the film is taken to be $N_W = 5.2x10^{22}$ $cm^{-3}$ derived from the average lattice-parameters (a-axis: 0.28117 nm and c-axis: 0.2815 nm obtained by XRD). The composition mentioned above and film thickness is determined by Rutherford backscattering spectrometry (RBS) of 1.8 MeV $^4He^+$ and 0.7 MeV $^3He^+$. Samples were exposed to deuterium-plasma generated by AC glow discharge of 1.5 KV in 67 Pa $D_2$ at room temperature, using the same method in [1]. Nuclear reaction, D($^3$He,α)P, analysis (NRA) was employed to detect D in the films and D-density was evaluated using α-particle yields.

Figure 1 shows NRA spectrum for a $WNO_{.4}$ film for D-plasma exposure time of 20 min (D fluence is estimated to be ~8x$10^{17}$ $cm^{-2}$), obtained by using 0.7 MeV $^3He^+$ ion beam (normal incidence and detection angle of 150 º ). α-particles emitted from the nuclear reaction are seen around 2 MeV and a broad peak around ~1.7 MeV represents protons with energies much smaller than the emitted energy (13 MeV) from the nuclear reaction, because the effective detection layer of the solid stated detector is far smaller to detect the whole energy of proton.

Using α-particle spectrum and W RBS yields, areal density of D integrated over the depth was evaluated and its dependence on D-exposure time is plotted in Fig. 2. It is seen that the areal density saturates at exposure time of 20m. The saturation value of 14x$10^{16}$ $cm^{-2}$ is remarkably larger than 2x$10^{16}$ $cm^{-2}$ for $WN_x$ (x~1) [2] and comparable with that in $WO_3$ [4]. Film thickness used in this study is 35 to 80 nm. No clear relation is observed between the areal density of D and film thickness, implying a considerable fraction of D's is retained near surface of films. D-plasma consists of 60 % of $D_3^+$ and 40 % of $D_2^+$ ($D^+$ can be negligible) [3]. If D's are assumed to be uniformly distributed within the projected range of 9.3 nm corresponding to the maximum energy of $D_2^+$ (2.12 keV), $N_D$/ $N_W$ yields ~3. Measurements of depth-distribution of D in $WNO_{.4}$ film, dynamic retention of D and thermal desorption of D are under way.

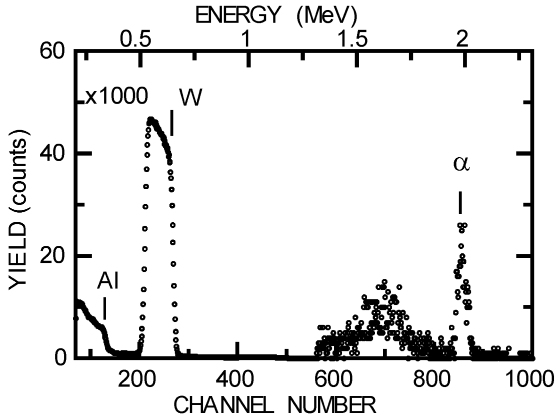


Fig. 1 NRA spectrum of α-particles (1.98 MeV) and protons (~1.7 MeV) for $WNO_{.4}$ film obtained by using 0.7 MeV $^3$He. D-plasma exposure time is 20 min. RBS-edge of W is seen at 0.66 MeV. Thickness of the film is 67 nm.

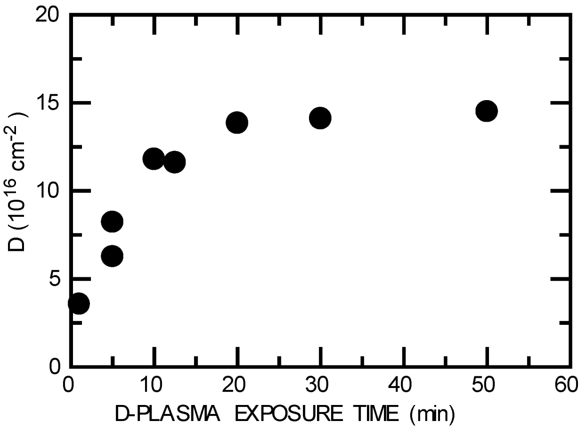


Fig. 2 D-density vs D-plasma exposure time.

1. Matsunami, N. et. al., Phys. Scr. T415(2011) 014042.
2. Gao, L. et. al., J. Nucl. Mater., 451(2014)352.
3. Sugai, H. et. al., J. Nucl. Mater. 128/129(1984)169.
4. Matsunami, N. et. al., J. Nucl. Mater. 390/391 (2009)693.