# Control of carbon agglomeration and hydrogen retention in low temperature plasmas with H-C-N reactive molecular system[2]

H-C-N反応性分子を有する低温プラズマ中における 炭素凝集と水素吸蔵の制御[2]

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Carbon dust particles and redeposited layer formed by plasma-surface interactions in fusion experimental devices retain large amount of hydrogen isotopes. Nitrogen injection into hydrogen plasmas has been used as one of the methods for suppression of carbon dust growth and reduction of tritium inventory. In this report, we have conducted experiments to investigate influences of nitrogen injection into  $H_2/CH_4$  low temperature plasma. From optical emission spectroscopy, volatile CN band spectra were observed in  $H_2/CH_4/N_2$  mixture plasmas. From mass spectrometry of  $H_2/CH_4/N_2$  mixture plasmas, volatile CN and HCN spectra were observed, which are key particles for suppression of the carbon dust formation.

## 1. Introduction

Dust particles are generated by plasma surface interactions, and contain large amount of tritium that is used as fuel gas in fusion reactors. Since carbon dusts retain large amount of hydrogen, the dust particles in fusion reactors bring several safety problems, such as tritium inventory and diffusion at an accidental air leakage. In addition, the dust particles containing tritium are transported into scrape off layer. The control of tritium inventory and suppression of dust particle release is important issues in future fusion reactors.

Nitrogen injection has been considered and tested as one of the methods for suppression of carbon dust growth and reduction of tritium inventory. In the experiments which are performed with  $Ar/H_2/N_2$ plasma irradiation to graphite targets, using high-power inductively coupled plasmas (ICPs), we got the results that nitrogen injection into  $Ar/H_2$ plasma leads to the significant suppression of carbon dust formation in the number of carbon dust particles[1,2].

In this paper, we have investigated effect of nitrogen injection into  $H_2/CH_4$  plasma in the experiments using Heliotron-DR, which generates low density and temperature, and pure  $H_2$  plasma in steady state condition. These conditions are near to remote plasma rather than ICPs's one.

### 2. Experimental setup

Low temperature RF plasmas with H-C-N reactive species are generated in hydrogen plasmas with small amount of  $CH_4$  and  $N_2$  injection.  $H_2$  gas

flow rate is 20 sccm. CH<sub>4</sub> and N<sub>2</sub> gas flow rate is about several sccm. The working gas pressure is ~1Pa. The electron temperature is 5-10eV. The electron density is  $(0.4-1.6) \times 10^{16} \text{m}^{-3}$  at the plasma edge. Four target stands for plasma irradiation are placed at different troidal sections. A silicon target of  $10 \times 10$  mm<sup>2</sup> is placed on a BN plate. Scanning Electron Microscope (SEM) is used to observe the generated dust particles on the targets. Energy Dispersive X-ray spectrometer (EDS) is also used to investigate the components of the dust and deposited layer. A film thickness meter is used to investigate the film thickness on the targets. Surface temperature of silicon is not actively controlled, but can be measured by a thermocouple which is placed behind. Mass spectrometry (QMS) and optical emission spectroscopy are used to observe the reactive species in the plasma with H-C-N system.

### 3. Results and discussion

Figures 1(a), (b) and (c) show calculation results of particle composition under thermal equilibrium plasmas. Figure 1(a) shows particle composition in C-H system. Figures 1(b), (c) show particle composition in C-H-N system. The composition of carbon containing particles widely changes between T=1000-3000K whether N<sub>2</sub> is injected or not. The density of high order carbon molecules like C<sub>4</sub>, C<sub>5</sub> are markedly decreases and the generation of volatile HCN molecule is remarkably enhanced as shown in Fig.1(c). These results show that atomic nitrogen in H-C-N plasma system hinders C-C combination necessary for nucleation in the carbon agglomeration process. In addition, generation of volatile molecules like HCN, NH is expected to contribute the removal of hydrogen isotope from the carbon deposit layer.



Fig.1 Calculation of particle composition in (a)C-H system (b)(c)C-H-N system

Following is the results of the experiments using Heliotron-DR. The surface temperature of silicon in the irradiation is  $T_s=320-370$ K. Figure 2 shows optical emission spectra of  $H_2/CH_4$  and  $H_2/CH_4/N_2$  plasmas. In this case, gas flow rates of  $H_2$ ,  $CH_4$  and  $N_2$  are 20, 5, 2 sccm, respectively. Irradiation time is 300 minutes. The molecular band spectra of volatile CN and NH radicals are clearly observed in  $H_2/CH_4/N_2$  mixture plasmas.

Figures 3 shows mass spectra of  $H_2/CH_4$  and  $H_2/CH_4/N_2$  mixture plasmas. In  $H_2/CH_4/N_2$  plasmas, mass spectra of volatile nitrogen particles like CN, HCN and NH increase strongly compared to those without nitrogen.

The carbon film thickness of silicon surface is 60-400 nm after 300 minutes  $H_2/CH_4$  plasma irradiation. On the other hands, it is 240-800 nm after  $H_2/CH_4/N_2$  plasma irradiation. In this case, the thickness of carbon film notably increases by nitrogen injection. From analysis by EDS, it is

found that the dusts are mainly composed of carbon. These results in preliminary experiments are different from those in ICPs experiments shown in 24P045-P.



## 4. Conclusions

Mass spectra and optical emission spectra show that generation of volatile molecules such as CN, HCN and NH by nitrogen injection into H<sub>2</sub>/CH<sub>4</sub> plasmas. These volatile molecules bring the suppression of carbon dust formation in the experiments using ICPs. In the first experiment using Heliotron-DR, this suppression effect was not seen. The difference between ICPs and Heliotron-DR experiments is the surface temperature of targets besides working pressure and so on. Carbon dust formation strongly depends on the surface temperature[2]. In this first experiment, the surface temperature is considerably low compared to ICPs's one. In order to confirm the suppression effect by nitrogen injection, using Heliotron-DR, higher surface temperature of targets may be needed.

#### References

- [1] M. Kyo et al: Plasma Fusion Res. 3, 2010.
- [2] M. Kyo et al: 19th Int. Symp. on Plasma Chem., Bochum, Germany, 2009.