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A study of carbon deposition layers on tungsten nano-structure
タングステンナノ構造表面上に形成された炭素堆積層の研究

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In order to investigate the effect of W nano-structure on co-deposition, co-deposition layers on nano-structure were prepared by magnetron sputtering and C/D mixed ion beam irradiation. In ion beam experiments, enhancement effect of nano-structure on carbon deposition was observed. However, in magnetron sputtering experiments, deposition rate were roughly the same between deposition layers on nano-structured and on mirror surfaces.

Formation processes of deposition layers were investigated by SEM and Raman spectroscopy. D retention properties were investigated by TDS.

1. Introduction

C and W are plasma facing materials for 1st set of ITER divertor. C has good thermal and mechanical properties, and low atomic number. However, C has a high sputtering yield and forms co-deposition layers with T. Therefore, studies on erosion and re-deposition behaviour of C are important for safety reasons in ITER.

Previous reports indicated that W nano-structure (fuzz) produced by high flux He plasma exposure enhanced C deposition, which was observed in TEXTOR tokamak [1]. However, there have been no systematic studies for deposition conditions, structure of deposition layers, and behaviour of hydrogen isotopes by precisely controlled experiments.

In this study, two types of carbon deposition experiments have been done; (1) irradiation by mixed D and C ion beam, (2) deposition by a magnetron sputtering method. Their structural properties were observed by SEM and Raman spectroscopy, and D retention properties by TDS (thermal desorption spectroscopy).

2. Experimental

W nano-structured samples were prepared by exposure to high density He plasma (flux: 1.3x10^{21} /m^{2}s, fluence: 4.7x10^{24} /m^2, bias voltage: -150 V). Initial thickness of nano-structure was about 500 nm estimated by SEM pictures.

Magnetron sputtering experiments were done at D_2 and Ar atmosphere. Ar and D_2 were introduced at the same flow rate by mass flow controllers and total ambient pressure was 20mTorr. A discharge voltage and a current were 450 V and 200 mA. Deposition time was varied from 45 min. to 24 h. Pure Ar atmosphere experiments were performed for comparison with Ar/D mixed experiments. In pure Ar experiments, a discharge voltage and a current were 550 V and 200 mA.

Ion beam experiments were done with high flux mixed ion beam device HiFIT[2]. Ion beams were generated with an incident energy of 150eV, an ion flux of about 1.0 x 10^{20} /m^{2}s, and an ion fluence of about 1.45 x 10^{24} /m^2. Carbon impurity in ion beams was introduced by putting carbon plates in an ion source chamber. Carbon impurity concentration was changed from about 1.0% to 3.2% by changing the number of carbon plate.

Raman spectra were observed in 800~2200cm^{-1} region. Raman spectra from all samples showed a broad G peak (at ~1570cm-^{1}), D peak (at ~1350cm^{-1}) and continuous background originated from photo luminescence. These features indicated the deposits were deuterated amorphous graphite. For data analysis, the G and D peaks were fitted with Gaussian after subtracting the PL signals also fitted with Gaussian curve.

For the TDS measurements, samples were heated by an IR heater with a ramping rate of 0.05 K/s. D_2 signal was calibrated by a D_2 calibrated standard leak and signals from other masses were estimated by using known sensitivity data of QMS.

3. Result

In ion beam experiments, threshold carbon concentration for C deposition on the mirror surface was about 4.0% at 473K, while C deposition on the nano-structure was observed around 1.5% C or more at the same temperature.

From SEM observation, the nano-structure were mostly sputtered by C ion bombardment in the low C concentration (1.0 %, without C deposition) at 473K and 723K. On the other hand, for experiments with higher C concentration than about 1.5 %, some of the nano-structure were sputtered, but its surfaces were covered with a C deposition layer.

Raman spectra of the C deposition layers by C/D mixed ion beam irradiation on the nano-structure had G peak positions at higher wavenumber than those of the C deposition layers on the mirror surface. However, the difference between these peak positions was small.

In magnetron sputtering experiments, regardless of deposition time, there were no significant differences in deposited mass between that on nano-structures and on mirror surfaces.

The deposition layers on the mirror surface showed laminar growth, while the deposition layers on the nano-structure initially showed island-like growth, see FIG.1. In this case, the height of these islands reached 800 nm after 3 h deposition. There were a lot of holes with diameters of 200~500 nm.

For longer deposition time, for example 7 h, the surface were completely covered with the C deposition layer. Since the initial deposition layer on the nano-structure had many pores, density of the deposition layer was lower than that on the mirror surface.

Raman spectra of the thick deposition layers on the nano-structure were similar to those on the mirror surface. However, Raman spectra of the thin deposition layers on the nano-structure (deposition time was 3 h) had different features as follows; (1) an intensity ratio between a D peak and a G peak (I(D)/I(G)) was larger than that on mirror surfaces.

(2) FWHM of a G peak was smaller than that of mirror surfaces. (3) A G peak position was shifted to lower wavenumber region. These features are commonly understood such that (1) hexagonal bond clusters decreased, (2) three dimensional disorder decreased, (3) sp3 bonds decreased. These trends were observed in the case of amorphous C films with relieved stress or with higher hydrogen retention [3,4]. These features of the initial C deposition layers on the nano-structure disappeared for the thick C deposit.

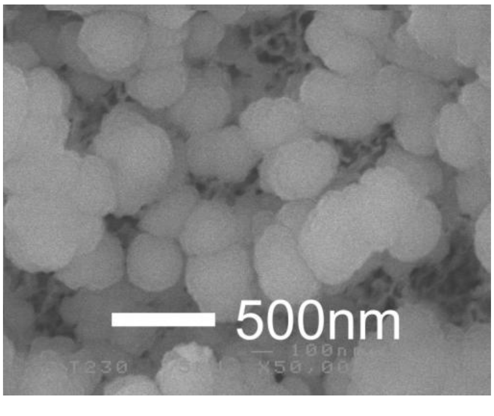

FIG.1 Surface image of deposition layer on nano-structure (3h sample)

For thin deposition layers (3 h deposition), total amount of released D_2 (mass 4) from the deposition layer on the nano-structure was less than that on the mirror surface by a factor of about three. Whilst the mass 20 signal from the deposition layer on the nano-structure was larger than that on the mirror surface. This difference in thermal desorption spectra suggested that retained D atoms in the deposition layer on the nano-structure tended to be released as hydrocarbon molecules. More detailed studies are in progress.

4. Summary

Enhancement of C deposition on the He induced nano-structure on tungsten was observed in C/D mixed ion beam irradiation experiments. Island-like growth of C deposition was observed in the early phase of deposition on the nano-structure in magnetron sputtering experiments.

The significant difference in Raman spectra between the deposition layers on the nano-structure and on the mirror surface was observed only in the early phase of deposition in magnetron sputtering experiments. TDS spectra showed retained D atoms in the deposition layers on the nano-structure tended to be desorbed as hydrocarbon molecules. More details will be presented.

References

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