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# STUDY ON PLASMA SURFACE INTERACTIONS FOR NUCLEAR FUSION

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## Abstract

Recent studies in Surface and Vacuum Science Laboratory, Hokkaido University are summarized. Experimental devices of our laboratory are also briefly introduced. Our main subjects are preparations and characterizations for coating films, surface modifications by plasmas and heat treatment, developments of low  $Z$  ceramics, ion irradiation effects on fusion first wall materials and plasma wall interactions in fusion confinement devices. For these items, surface analysis techniques and ultrahigh vacuum techniques are effectively utilized. Our devices typically used are AES, XPS, TDS, RS, SRF, EBE, SEM, magnetron and RF sputterings, a cylindrical plasma device and ECR ion source. For the plasma surface interaction study, our research work is aimed at the task in which both simulation and actual confinement plasma experiments are performed. Our current activities on plasma surface interactions are briefly described.

## 1. Introduction

Our group was established in 1969 in Department of Nuclear Engineering, Hokkaido University. Since then, we have studied on applied fields that require ultrahigh vacuum techniques, and simultaneously we have developed surface analysis techniques to understand the surface phenomena. In 1975, fusion material program and plasma surface interaction study were initiated to utilize our developed analysis techniques. These studies have been successfully performed so far. In addition, surface modification<sup>1)</sup> studies based on coating preparation and surface analysis devices have progressed. We now have five educational/research staff members, a secretary, four graduate students and five undergraduate students.

In the following, our research summary, experimental devices and recent activities are described.

## 2. Research Summary

- 1) Interactions of low  $Z$  materials with hydrogen ions
  - a) irradiation effects of hydrogen ions on graphite, ceramics such as TiC, SiC and B<sub>4</sub>C,

- and carbon coating films
- b) development of high current ECR ion source
  - c) chemical sputterings of fusion first wall materials
- 2) Preparation and characterizations of low Z ceramic coatings
    - a) Ti coatings on stainless steel and its surface modification by heat treatment
    - b) preparation of C+TiC coatings and ion irradiation effect
    - c) ion irradiation effect on carbon coatings prepared by EBE
  - 3) Surface properties of alloy
    - a) change of surface compositions of B<sub>4</sub>C
    - b) selective sputterings and segregations of Co-Cr<sup>13)</sup> and Nb-C
  - 4) Carbon coating films
    - a) carbonization experiments in Heliotron E, TEXTOR tokamak, JIPPT-IIU tokamak, ECR-II(RIKEN) and plasma simulator device
    - b) characterizations for carbon coatings, such as erosion by hydrogen ions and hydrogen content of the film
    - c) optimization for carbon coatings as fusion first wall material
  - 5) Characterizations of nuclear grade graphite as fusion first wall material
    - a) vacuum engineering properties such as gas desorption and internal surface area
    - b) effects of hydrogen ion bombardment by ECR ion source
    - c) thermal mechanical properties
    - d) surface modifications by coatings
    - e) new carbon material such as C/C composite
  - 6) Getter material development
    - a) method to pump hydrocarbon from vacuum system
  - 7) Boundary plasma physics
    - a) impurity and recycling particle measurements
    - b) interactions of edge plasmas on first wall material

### 3. Experimental Devices

For the surface analysis, the following devices are used ;

- 1) Auger electron spectroscopy (AES) for depth compositions of coatings and alloy surface
- 2) X-ray photoelectron spectroscopy (XPS) for chemical binding state and accurate atomic composition
- 3) X-ray diffraction (XRD) analysis for crystal structure
- 4) scanning electron microscope (SEM) for surface morphology
- 5) thermal desorption spectroscopy (TDS) for gas desorptions
- 6) interferometer (Transky method) for film thickness
- 7) surfrecorder for surface roughness

For the preparations of coating films,

- 1) magnetron sputtering
- 2) electron beam evaporation
- 3) RF sputtering
- 4) RF/DC plasmas in cylindrical chamber (Q machine type device)

are used. To investigate the ion irradiation effects on low Z materials, the following ion sources are used.

- 1) ECR ion source
  - 2) duoplasmatron ion source with a chamber in which in situ TDS analysis is performed.
- The devices quite frequently used are briefly described below.

The AES with electron image is mainly used to analyze the depth composition profile. One of our AES devices has a heating structure for samples, so that in situ analysis for the

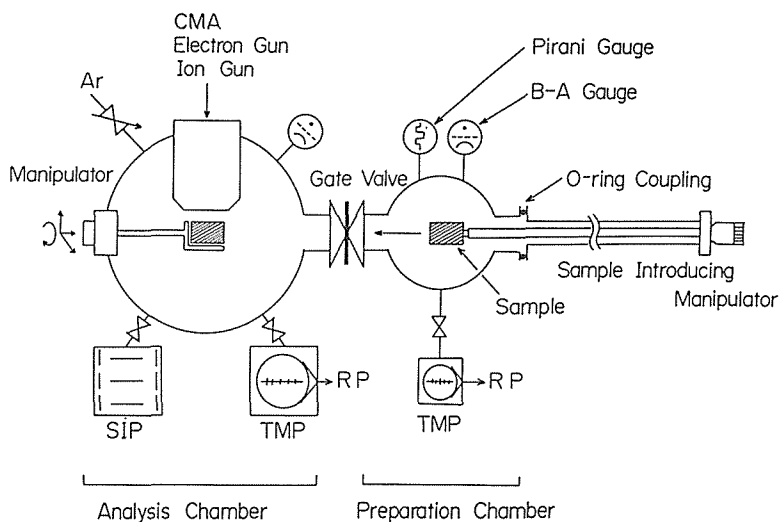


Fig. 1 Schematic view of AES device

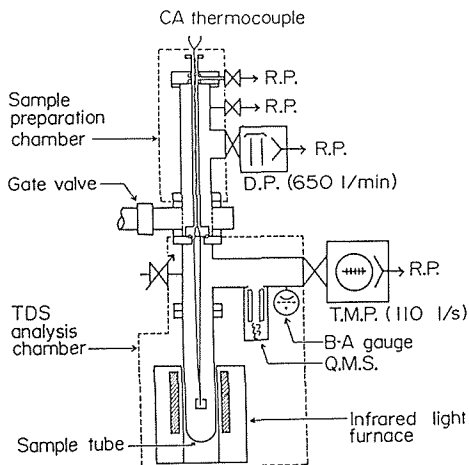


Fig. 2 Thermal desorption spectroscopy (TDS) device

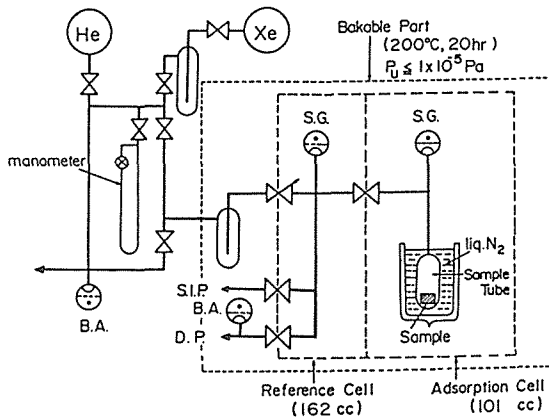
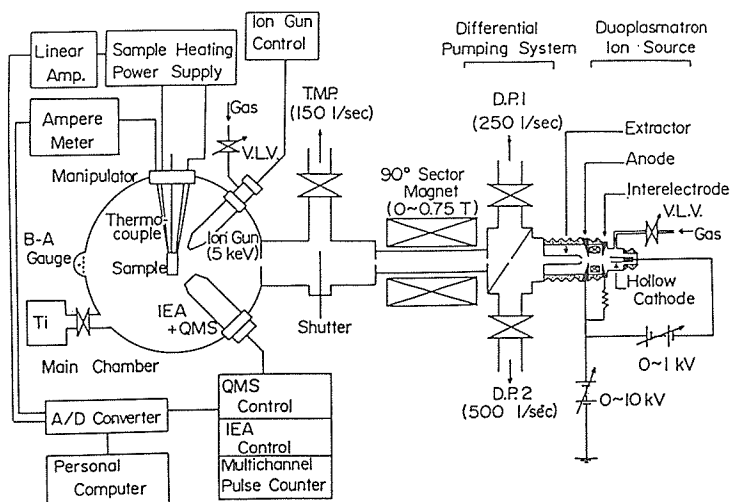
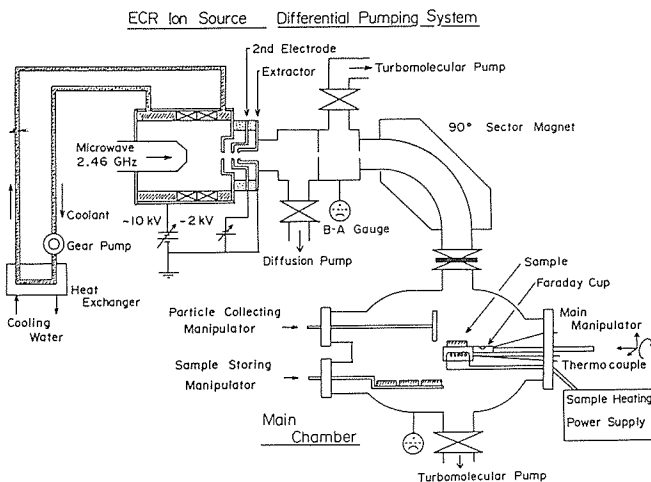


Fig. 3 Surface roughness factor (SRF) measurement device



surface composition during the heating phase can be done. Figure 1 shows the AES device that is an air lock type.

The TDS and the surface roughness factor (SRF) measurement devices shown in Figs. 2 and 3 measure the released gas amount and the ratio of micro surface area to geometrical area, respectively. In the TDS, sample temperature is raised from room temperature to 1,000°C. The desorbed gas is analyzed by QMS. The SRF device consists of an adsorption cell and preparation cell. For the adsorption, Xe gas is used.

The ECR ion source consists of ECR plasma source, sector magnet and irradiation chamber (Fig. 4). Typical fluence of hydrogen ions is  $10^{19}/\text{cm}^2$  for several hours operation. Duoplasmatron ion source has a chamber in which in situ TDS analysis is available (Fig. 5). So the irradiation effect on the gas desorption is easily analyzed. The plasma simulator device shown in Fig. 6 is used to make carbon coating films under variable operation

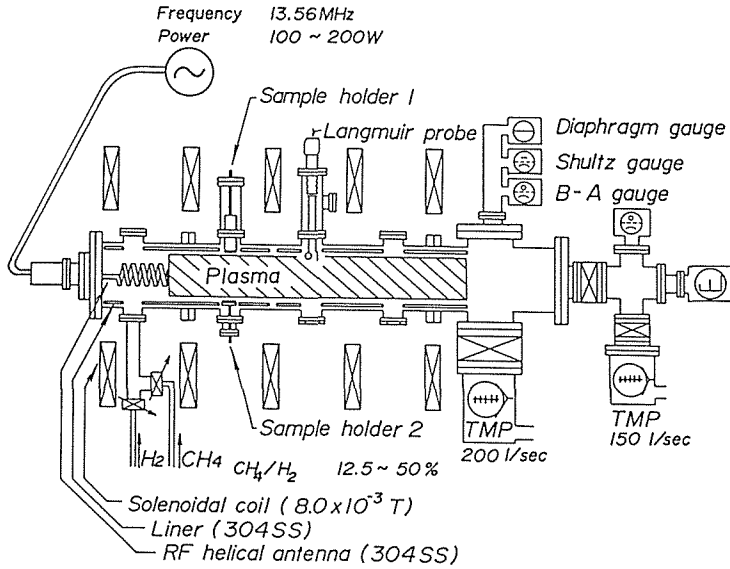


Fig. 6 Plasma simulator device (Q machine)

conditions. Now the optimization study for the film as a fusion first wall is performed.

We also have collaborative studies with teams of Heliotron E (Kyoto Univ.), ECR-II (RIKEN) and JIPPT-IIU (Nagoya Univ.). In Heliotron E, schematic carbonization study is now underway. In particular, the erosion rate of the film due to fusion plasmas and the redeposition rate have been investigated.

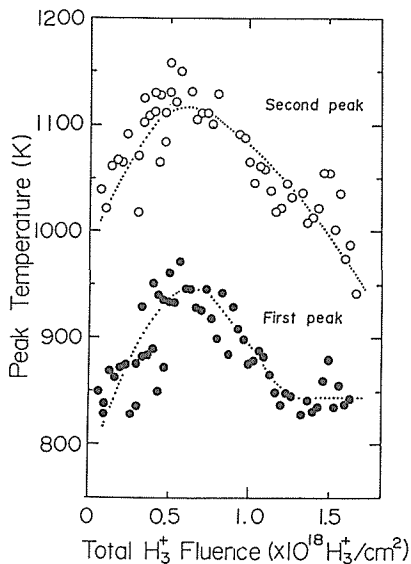


Fig. 7 Change of peak temperature due to total ion fluence (irradiation number  $\times$  fluence per one irradiation)

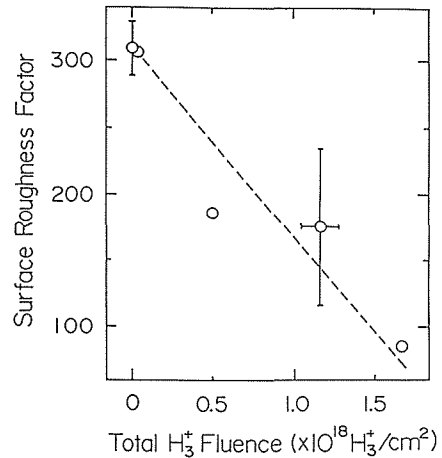


Fig. 8 Change of surface roughness factor due to hydrogen ion irradiation

#### 4. Recent Activities

##### 1) Gas desorptions and surface roughness due to hydrogen ion irradiation<sup>2)</sup>

The thermal desorption process and surface roughness of POCO graphite irradiated by hydrogen ions were investigated. The energy of the ion was 1.5 keV, and the desorptions of hydrogen and methane were measured by the TDS. The surface roughness after the irradiation was also measured by BET method. The trapped hydrogen was desorbed in forms of H<sub>2</sub> and CH<sub>4</sub>. The ratio desorbed in the form of CH<sub>4</sub> is 1-2%. The activation energy of methane was 2.3 eV. In the TDS analysis of hydrogen, two peaks of the desorption curve were observed. The activation energy of hydrogen was 1.2 eV. The change of the peak temperatures in the hydrogen desorption curve due to the irradiation number was also observed (Fig. 7). As seen in Fig. 8, the surface roughness was reduced with the increase of hydrogen fluence. The relation of the surface roughness with the gas desorption was discussed.

##### 2) Measurement of hydrogen and impurities deposited on surface probe in Heliotron E<sup>3)</sup>

In the edge plasma of Heliotron E, a-Si and ZrD<sub>x</sub> probes were placed and the retained hydrogen and impurities were measured by TDS. Nuclear Reaction Analysis was used to determine the radial profile of the retained hydrogen. The retained hydrogen amount increased with the plasma density. The radial profile was approximately flat. During the neutral beam injection, the impurities deposited on the probe were measured. These amounts increased with the beam power. The impurity species were Fe, O, Cr and Ni. Thus, the retained hydrogen and impurities were successfully measured by the present probe technique.

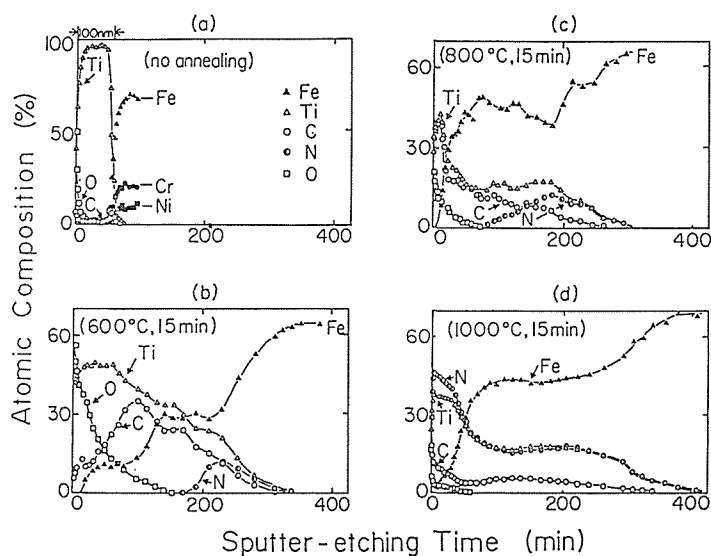


Fig. 9 Change of surface composition by heat treatment with various temperature

### 3) Surface modification of stainless steel by Ti-coatings<sup>4)</sup>

The stainless steel substrate was coated by Ti in a magnetron sputtering device, and the sample was heated at 600–1,000°C. The change of the surface composition was analyzed by AES and XPS. The formations of TiC and TiN were observed after the heat treatments of 800°C and 1,000°C, respectively (Fig. 9). The hardness was measured by Knoop hardness tester. The maximum hardness was 2,900 kg/mm<sup>2</sup>. Thermal desorptions for Ti-coated stainless steel was also analyzed by the TDS. The desorptions of H<sub>2</sub>, CH<sub>4</sub> and N<sub>2</sub> were considerably suppressed compared with the stainless steel without the Ti coatings.

### 4) Surface composition change of B<sub>4</sub>C by heat treatment<sup>5)</sup>

The B<sub>4</sub>C single crystal sample was resistively heated in AES device, and in situ AES analysis was done to examine the surface composition change. The heating temperature was from 600°C to 1,200°C. The boron concentration decreased with the heating time and then attained a steady state. The steady state value decreased with the temperature for a range less than 1,000°C but increased in the bulk concentration at 1,200°C. The evaporated particles from the sample were measured by collection probe. It was observed that only boron was evaporated from the surface. A simple analytical model to explain the composition change was

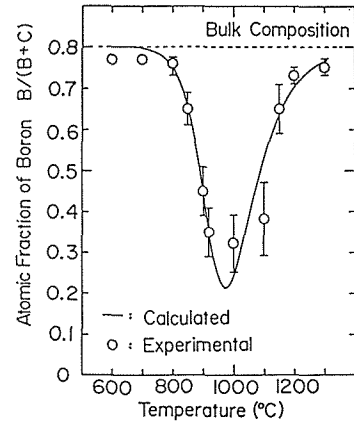


Fig. 10 Change of surface composition in term of heating temperature, solid line shows the calculated curve

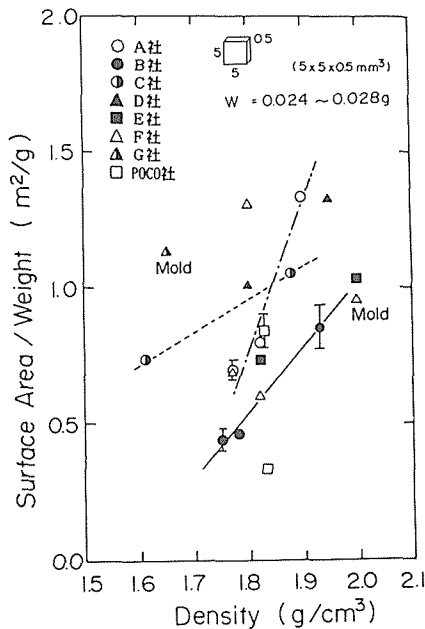


Fig. 11 Surface area of graphite versus bulk density

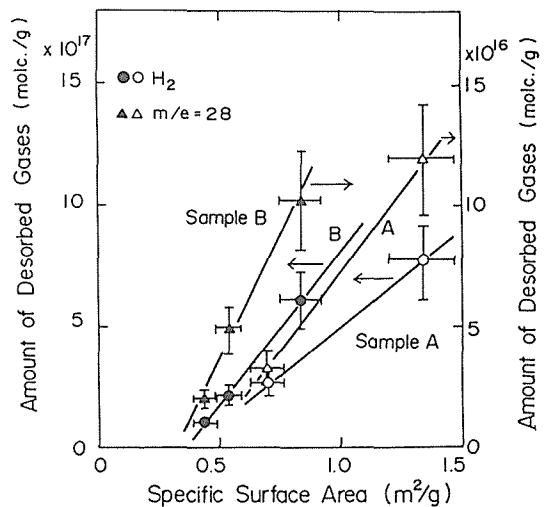


Fig. 12 Amount of desorbed hydrogens versus surface area



proposed. This model can explain the change of the surface composition by heat treatment (Fig. 10).

### 5) Characterizations of graphite as fusion first wall material<sup>9,10)</sup>

Eighteen graphite materials were gathered from seven major graphite companies in Japan. The properties regarding surface and vacuum engineering were examined. It was found that the released gas amount and the surface roughness strongly depended on the production process and the final treatment. However, for samples made by similar processes, both the gas desorption amount and the surface roughness increased with the bulk density. The surface area per gram versus the density and the desorbed hydrogen amount versus the surface area are plotted in Figs. 11 and 12, respectively. The hydrogen ion with an energy of approximately 1.5 keV was used to examine the irradiation effect on the graphite surface. By the irradiation, the surface became smoother and at a temperature of 500°C, many small dips appeared.

The properties of the gas release and the surface roughness were related to the plasma discharge conditions and then the required conditions for the graphite as the fusion first wall material were analyzed.

### 6) Preparations of carbon coatings and characterizations<sup>6-8,11,12)</sup>

The carbon coating films produced in TEXTOR, JIPPT-IIU, ECR-II, Heliotron-E and our plasma simulator devices were characterized mainly by AES and TDS. For the sample without heating, the hydrogen content was from 30 to 50%. In the plasma simulator device, the sample was heated during carbonization. The hydrogen content was greatly reduced in this case. The erosion rate of this film due to energetic ions was also small. The optimized film that showed a low erosion rate and low hydrogen content is now studied. Figure 13 shows the etching rate and the hydrogen content of the carbon films produced in several

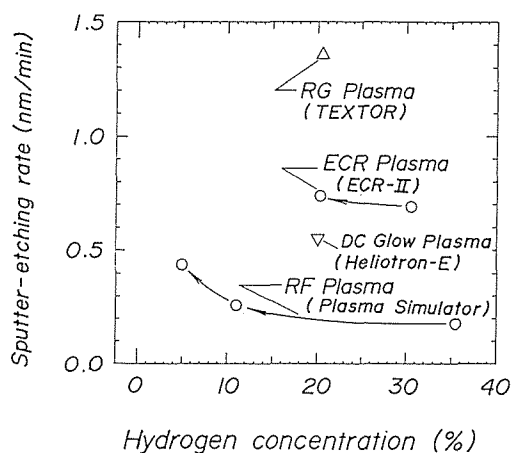


Fig. 13 Hydrogen content and etching rate due to ions of carbon films produced in various discharge

devices.

In order to suppress the chemical erosion, TiC or C+SiC ceramics have been used as the first wall material. If the content of Ti or Si is large, the radiation loss from the fusion plasma is enhanced because of the higher atomic number. Thus we tried to produce C+TiC film with a low content of Ti. For the film with Ti/C=0.2, the hydrogen ion was injected and the change of the film thickness was measured. Compared with the case of pure carbon film, the present film showed much less reduced erosion rate.

In the present large tokamak, the Inconel 625 vacuum chamber is often used. To apply the carbonization, we first consider the life of the carbon film under a condition of high temperature and high heat flux. When the carbon coated Inconel was heated at 700°C, the segregation of Cr was observed. It was found that Cr-carbide was formed in the film region and this played a role of a diffusion barrier against the carbon. Therefore, the Cr-carbide is effective to lengthen the life of the carbon film. The film was also exposed to hydrogen ions, and the resultant erosion rate was comparable with that of bulk graphite. We now are developing the method to suppress the erosion by combining an additional material layer.

## 5. Summary

In our laboratory, we have developed surface analytical techniques and ultrahigh vacuum techniques. These processes have been applied to the fields in which both techniques are important; plasma wall interactions in fusion device, new ceramics developments and surface modifications by plasmas. Our established technology can be also applied to new field such as space shuttle material development and vacuum diagnostics in the wake of the shuttle. We continuously challenge for new subjects to effectively apply our surface and vacuum technology.

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