

## Tokamakium in TRIAM-1M and its Impacts on Plasma-Surface Interaction

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### Abstract

Tokamakium in TRIAM-1M was consisted of very fine defective Mo crystals; the grain size was about 1nm and the crystalline structure is not normal bcc but fcc-like because of co-deposition with oxygen. Due to the co-deposition, retention of hydrogen isotope is changed considerably. Thermal desorption of implanted deuterium indicates that the retention is much larger and stronger than that of normal Mo. The defective structure and the micro-cavities contribute to the large hydrogen retention.

### Keywords:

TRIAM-1M, tokamakium, deposit, microstructure, hydrogen retention, plasma-wall interaction

### 1. Introduction

Inner walls of plasma confinement devices are more or less covered by the deposits of impurities emitted from the plasma facing materials due sputtering and/or evaporation. Such phenomena have been observed in many tokamaks and the deposited material is named "tokamakium" [1-8]. Especially in carbon dominant devices, strong co-deposition of carbon with hydrogen isotope was observed [2-4]. Formation of the tokamakium changes physical and chemical properties of the plasma facing surface. Particularly, change of hydrogen retention properties may have large impacts on hydrogen recycling under long pulse operation.

In the present work, therefore, micro-structure of the tokamakium in TRIAM-1M was observed and its properties concerning with hydrogen retention were examined by using comparable vacuum-deposited material.

### 2. Experiments

TRIAM-1M is a superconductive high field tokamak with a vacuum vessel made of 304SS, divertor plates of Mo and limiters of Mo. Non-metallic materials

such as graphite have never used for in-vessel components. To examine the surface modification by impurity deposition, impurity collectors (pre-thinned 304SS disks) mounted on the head of a surface probe system [9] were exposed to the high-ion temperature mode plasma [10] at the electron drift side (E-side) in the scrape off layer (SOL). Duration of each discharge was about 1 minute and the total exposure time was 31.5 minutes. Typical plasma parameters were following:  $I_p = 20\text{--}25$  kA,  $n_e \sim 2 \times 10^{12}$  cm<sup>-3</sup>,  $T_i = 1.5\text{--}2.5$  keV. After the exposures microstructure and composite elements of the deposits on the thin 304SS disks were examined with a transmission electron microscope (TEM) with EDS. By using dark-field imaging technique one can obtain the image of the deposits without any serious effects of the substrate.

According to the result that major metallic elements of the tokamakium in the TRIAM-1M was Mo [7,8], vacuum-deposition experiments of Mo were carried out in various atmosphere to simulate it. Taking into

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account that major gas components during a plasma discharge is hydrogen and in addition some oxygen and water exist as residual impurities, the vacuum-depositions were carried in a high vacuum ( $<10^{-4}$  Pa) and low-pressure hydrogen or oxygen atmospheres. Partial pressures of hydrogen and oxygen were  $P_{H_2} = 10^{-3}$  Pa and  $10^{-2}$  Pa,  $P_{O_2} = 5 \times 10^{-4}$  Pa,  $5 \times 10^{-3}$  Pa, respectively. Microstructure of the deposits was examined with a TEM.

Mo-deposits formed in oxygen atmosphere ( $P_{O_2} = 5 \times 10^{-3}$  Pa) were implanted with 6 keV- $D_3^+$  deuterium ions (2 keV- $D^+$ ) at room temperature up to a fluence of  $1 \times 10^{22}$  ions/m<sup>2</sup>. It is likely that the majority of the implanted ions were stopped within the deposit, because its thickness of about 50 nm was larger than the projected range of 2 keV- $D^+$  ions ( $<30$  nm). Thermal desorption of molecules composed of deuterium (DH,  $D_2$ , DHO,  $D_2O$ ) under heating with a ramping rate of 1K/s was measured with quadruple mass spectroscopy (QMS). QMS signals of DH,  $D_2$  and DHO,  $D_2O$  were quantified by using He standard leak. Thermal desorption spectra from the bulk Mo (99.95% purity) and bulk 304SS were also obtained as references.

### 3. Results and Discussion

#### 3.1 Microstructure of the deposits

Figure 1 shows dark field images and corresponding electron diffraction patterns of the tokamakium deposited on a thin 304SS specimen in TRIAM-1M. The images were obtained from a part of the first broad diffraction ring. At this imaging condition, only the crystal grains satisfying the Bragg

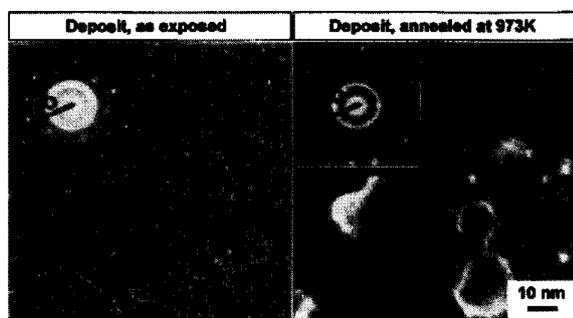


Fig. 1 Electron diffraction patterns and microstructures of the deposition on the stainless steel specimen exposed to tokamak discharges (left) and that annealed at 973 K for 10 minutes (right). White image in these micrographs shows individual crystal grains.

diffraction condition have white image. Numerous small dot images indicate that the deposit is consisted of fine crystal grains around only 1 nm in diameter. The radius and intensity of the diffraction rings indicate that crystal structure is fcc-like, though its major metallic component is Mo, which normally form bcc structure. One should note that the crystal structure must be very defective in such fine grains, because more than half of the constituting atoms is expected to locate on or near the grain boundaries. As shown in the picture (right) of Fig. 1, thermal stability of the deposit is quite low. Grain growth and simultaneous change of crystal structure from fcc-like to bcc occurred abruptly by the isochronal annealing (100 K steps for 10 minutes) up to 973 K. Diffraction pattern shows no oxidation. These results indicate that the structure and the characters of the tokamakium formed in TRIAM-1M is completely different from the normal vacuum-deposited Mo, which are polycrystalline consisted of bcc grains of about 200 nm in size [8].

As reported in ref. [8] residual oxygen strongly affects structure of vacuum-deposited Mo. If the ratio of the arrival rate of oxygen atoms and Mo atoms,  $A_{O}/A_{Mo}$ , is about 2–3, its structure is normal large bcc grains while if the ratio is about 20, fine fcc-like grain structure, similar to the deposit of TRIAM-1M, appear. It is likely that oxygen atoms adsorbed on the surface suppress free migration and crystallization of the deposited Mo atoms and result in refinement of the crystal grains. Moreover, oxygen atoms involved in the Mo lattice may prevent to form normal bcc structure and cause distortion of lattice due to its large atom size. Similar fcc-like fine grain deposit was observed in Fe formed in oxygen atmosphere [11]. It was shown that the structure changed successively from large bcc grains, fine fcc-like grains, fine FeO grains to fine  $Fe_2O_3$ - $Fe_3O_4$ , with increasing partial pressure of oxygen during the vacuum-deposition.

Hydrogen atoms, in contrast, did not show strong effects on the structure of the vacuum-deposited Mo. The structure is large bcc grains even if the ratio  $A_H/A_{Mo}$  is about 200 [8]. Weaker chemical interaction with Mo and its smaller atomic size may reasons why the effects of hydrogen is not so strong as oxygen.

Though TRIAM-1M is an ultra-high vacuum machine, some oxygen exist as residual impurity. It is likely that co-deposition of the residual oxygen with Mo brought the peculiar defective structure.

### 3.2 Hydrogen retention in the defective Mo deposition

Due to their defective structure, large and strong retention of hydrogen is expected for the tokamakium in TRIAM-1M and the Mo-deposits formed in a low pressure oxygen atmosphere. To estimate the retention of deuterium, implantation of deuterium ion was carried out. Figure 2 shows thermal desorption spectra (TDS) of  $D_2$  ( $m/e = 4$ ) and  $D_2O$  ( $m/e = 20$ ) from the deuterium ion implanted Mo-deposit, which was formed in the atmosphere of  $5 \times 10^{-3}$  Pa  $O_2$ . TDS data for the bulk Mo and 304SS are also plotted in the figure. In case of  $D_2O$ , large desorption occurred only for the Mo-deposit. At least four peaks exist above 450 K and desorption still continues even at 900 K. This large desorption indicates that quite a large amount of oxygen are involved in the defective Mo-deposit.

As shown in Fig. 2, retention of implanted deuterium in the Mo-deposit is much larger and much stronger than bulk Mo. For the Mo-deposit, two large desorption peaks of  $D_2$  are distinguished at around 550 K and 720 K, while only a small peak at around 530 K for the bulk Mo. The thermal desorption spectrum from the Mo-deposit is also different from that of bulk 304SS. This indicates that majority of the implanted deuterium are retained in the deposited layer.

Retention of deuterium in deuterium ion implanted Mo-deposit, bulk Mo and 304SS are plotted in Fig. 3 against deuterium ion fluence.

Each data is the sum of the desorbed deuterium as  $DH$  and  $D_2$ ,  $DHO$  and  $D_2O$  molecules. In contrast to the weak retention in bulk Mo, the defective Mo-deposit shows strong retention. It is worth to note that trapping of deuterium in the Mo-deposit is even larger than stainless steel, which is known as a strong hydrogen trapping material. The ratios of  $D/Mo$  and  $O/Mo$  at the fluence of  $1 \times 10^{22}$   $D/m^2$  were estimated to be 0.35 and 0.04, respectively.

Microstructure of Mo-deposit implanted 4 keV- $H_2^+$  hydrogen ion at room temperature to a fluence of  $7.2 \times 10^{21}$   $H/m^2$  is shown in Fig. 4. The micrographs are bright field images taken at just-focus, under-focus and over-focus conditions, respectively. The white image in the under-focus condition reverses to black one in the over-focus condition as indicated by arrows in the figure. Such reversible change of the image contrast indicates that the defects are micro-cavities. It seems that the defective structure and rather easy formation of small cavities are the reasons for large and strong retention of hydrogen.

Figure 5 is a micrograph at low magnification of the Mo-deposit implanted 4 keV- $H_2^+$  hydrogen ion at room temperature to a fluence of  $7.2 \times 10^{21}$   $H/m^2$ . The substrate was an Al thin foil. It is clear that the

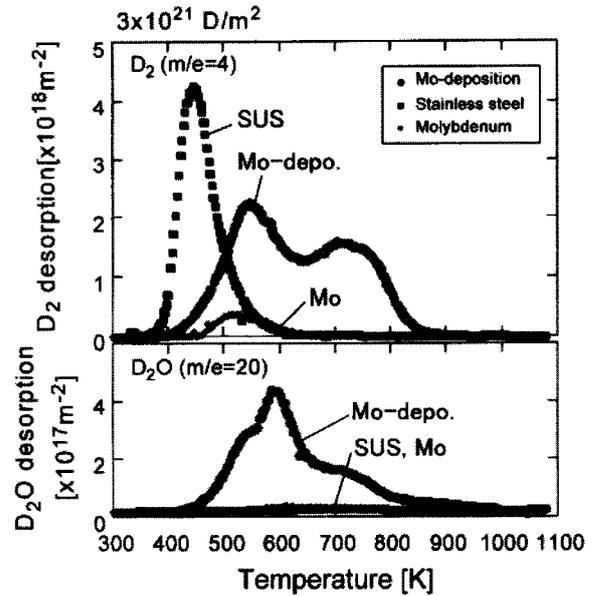


Fig. 2 Thermal desorption spectra of  $D_2$  ( $m/e = 4$ ) and  $D_2O$  ( $m/e = 20$ ) obtained from the Mo-deposit in oxygen atmosphere ( $P_{O_2} = 5 \times 10^{-3}$  Pa), Mo and 304SS specimens implanted with 6 keV- $D_3^+$  at a fluence  $3 \times 10^{21}$   $D/m^2$ .

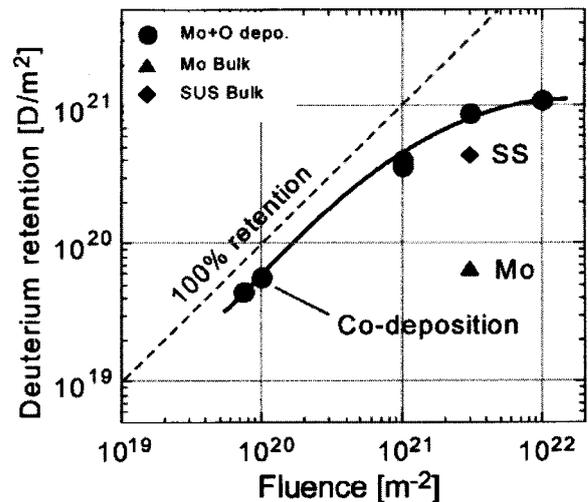


Fig. 3 Fluence dependence of the deuterium retention for Mo-deposit. Deuterium retention in bulk Mo and 304SS are also plotted. Specimens were implanted with 6 keV- $D_3^+$  at room temperature before the TDS experiments.

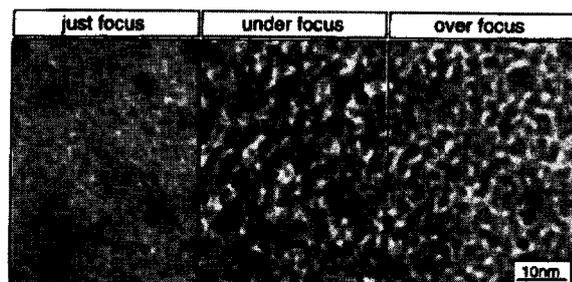


Fig. 4 Microstructure of Mo-deposit implanted with 4 keV-H<sub>2</sub><sup>+</sup> at room temperature to a fluence of  $7.2 \times 10^{21}$  Hm<sup>-2</sup>. Each micrograph was taken at the conditions of just-, under- and over-focus.

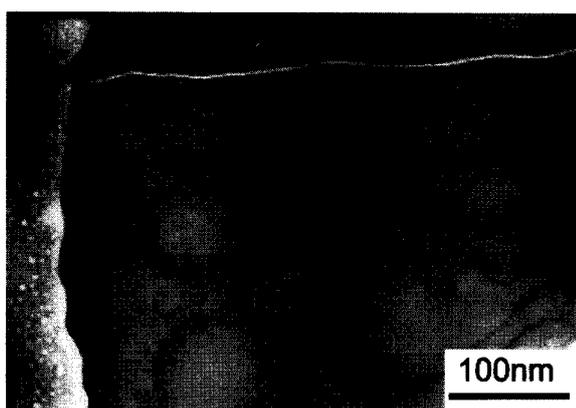


Fig. 5 Blisters formed in Mo-deposit by hydrogen ion implantation at room temperature.

deposited layer form blisters by the accumulation of implanted deuterium at the interface between the substrate and the deposit layer. Similar blistering was observed in the tokamakium formed in TRIAM-1M [7].

Present work indicates that re-deposition of sputtered impurities on plasma facing surface causes serious problem such as large retention of hydrogen isotopes even in metallic tokamak as well as carbon tokamak [2-4].

#### 4. Conclusions

A major element of the tokamakium in TRIAM-1M is Mo but its structure is peculiar; crystal grain size is about 1nm and their crystal structure is not normal bcc but fcc-like because of co-deposited oxygen, which exists in plasma as residual impurities. The Mo-deposits formed in the low pressure oxygen atmosphere, which have the same structure of the deposit in TRIAM-1M, show very large and strong hydrogen retention. Implanted deuterium is desorbed not only as D<sub>2</sub> and DH

but also as D<sub>2</sub>O and DHO. The defective structure and the micro-cavities contribute to the large hydrogen retention. The ratio of D/Mo reached 0.35 at high fluence.

The present work indicates that re-deposition of sputtered elements on plasma facing surface is a serious problem even in a metallic tokamak. Due to its defective structure and involved oxygen, the deposited layer must trap large amount of hydrogen and may affect hydrogen recycling. One should pay more attention to the modification of plasma facing surface by the impurity deposition and its effects on the hydrogen behavior. This is particularly important for long pulse and steady state plasma.

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