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Siliconization of Molybdenum Using Glow Discharge Method

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ABSTRACT

In the first application of a low-Z protective material to a fusion reactor, a thin layer of silicon film was deposited on molybdenum samples at room temperature using a mixture of silane and He gas (90 % He+10 % SiH4) using a glow discharge method. Siliconization was conducted for 170 min with the gas pressure of 4–6 $\times 10^{-2}$ torr. The DC power supply parameters were as follows, the anode voltage of 500–600 V and the current of 190-210 mA. The films have been characterized using scanning electron microscopy, EDX, X-ray diffraction, FTIR, and AFM. SEM images show that the silicon coating covers the entire surface smooth, and there is no boundary for granulation. Based on AFM measurements, the coating thickness was estimated to be 450 nm. This study used FTIR to detect the Si-O-Si tensile vibrations and the Si-H tensile vibrations.

Keywords**:** Low Z Material; Molybdenum, Silicon Coating; Glow Discharge.

1. Introductions

Using high atomic number materials as components in contact with plasma can cause many plasma instabilities [1]. In the presence of high-Z ions that penetrate the last closed magnetic surface and are transported into the plasma core, intense energy losses occur from the plasma core. [2]. As a high z material, Molybdenum has high melting points, good thermal conductivity, and acceptable thermomechanical properties, which is a candidate material as a limiter or diverter [3].

The low sputtering yield of tungsten and molybdenum PFCs has been exploited to produce clean core plasmas. A tungsten-coated molybdenum poloidal limiter was tested in the FTU tokamak [4]. Boronization in the Mo-walled Alcator C-Mod tokamak leads to a considerable decrease in the molybdenum core radiation and better H-mode performance [5].

Alcator and Frascati tokamaks have always employed high-Z materials such as iron, nickel, and molybdenum as limiters or diverter plates. In the Frascati Tokamak Upgrade (FTU), a series of experiments have been performed in which the limiter material was changed from Inconel to molybdenum (TZM) and tungsten [2]. The limiter of HT-7 was made of molybdenum before 2000 [6]. During the first plasma experimental campaign of EAST, its limiters were Molybdenum [7]. Also,

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metallic coatings usually increase the oxidation resistance of Mo-based alloys. The deposition of the silicon layer was found to have a beneficial impact on the oxidation resistance of Mo-based alloys [8].

2. Experimental

The experimental setup of the glow discharge conditioning system is represented schematically in figure 1. Mo samples were prepared in 1×5 cm specimen. The molybdenum sample has been placed at the wall surface using a sample holder, as presented in Fig. 1, and it has been electrically insulated from the vessel wall. Silicon coating is deposited onto Mo samples using the glow discharge method.

He-glow discharge was performed for several hours before siliconization. Then a gas mixture of 90%He+10%Si was injected into the chamber through the mass flow controller. The GDC parameters were as follows, the gas pressure of 4–6 $\times 10^{-2}$ torr, the anode voltage of 500–600 V, and the current of 190-210 mA. Siliconization is conducted due to 170 minutes. Structural analysis of the coating has been investigated using SEM, EDX, profilometer, and FTIR.

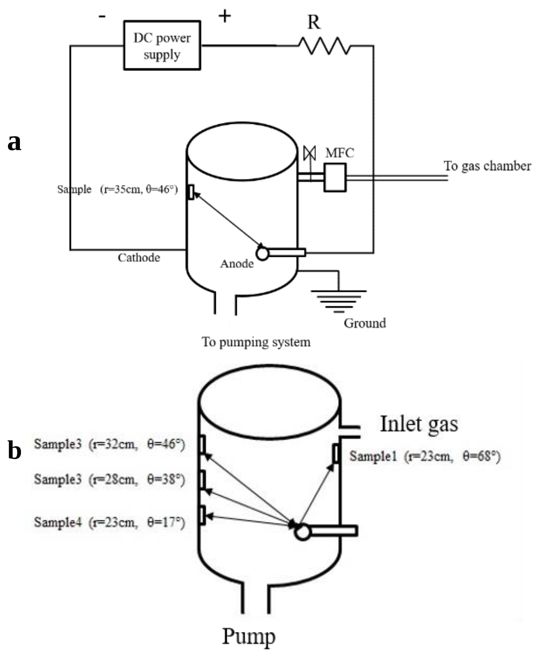

Fig.1. a. The position of the samples on the wall relative to the anode, **b.** Schematic diagram of glow discharges conditioning setup.

3. Results and Discussion

Scanning electron microscopy and energy-dispersive X-rays were conducted to investigate the coating morphology and elemental analysis of the coating. The SEM images of the samples are shown in Figure 2. SEM images show that the coating covers the entire surface relatively evenly. Despite the high magnifications, the coating does not show any granulation boundaries and covers the surface uniformly. In sample 1, some cracks with a length of 1 micron are observed, either due to the preparation of the sample before coating or cracks that have been created during the coating process (Fig. 2).

The EDX analysis of the samples is shown in Figure 3. Table 1 shows the elemental analysis of siliconized molybdenum samples at different depths of the coating surface. As can be seen from Table 1, as the accelerator voltage decreases, the penetration depth of the electron beams into the surface decreases, and the percentage of silicon increases, indicating that a silicon layer has formed on the surface.

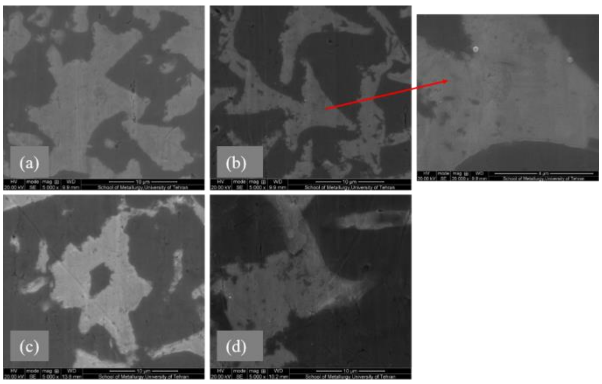

Fig.2. SEM images of 170 minutes siliconized molybdenum samples; **a.** Sample 1, **b.** Sample 2, **c.** Sample 3, **d.** Sample 4.

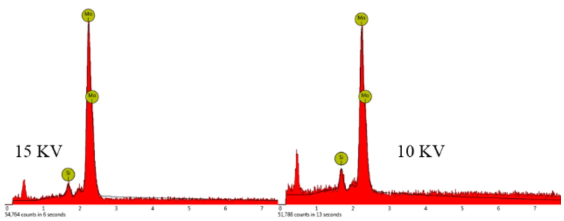

Fig.3. EDX analysis of siliconized sample during 170 minutes.

Table 1. Elemental analysis of siliconized molybdenum sample at different depths of the coating surface.

Accelerated voltage	Element	
	Mo	Si
15 kV	93.4	6.6
10 kV	89.1	10.9

To measure the coating thickness, the surface of the samples is scanned using AFM. During the siliconization process, a part of the sample was covered using a mylar layer which creates a step to measure the coating thickness against the uncoated surface. Figures 4 and 5 show the coating thickness of one siliconized molybdenum sample in approximately 170 minutes. As presented in Fig. 4,minor variations in vertical stylus displacement are depicted as a function of position. The coating thickness is around 450 nm, as displayed in Fig. 4. Xrd Analysis of siliconized Mo sample is shown in Figure 6. The presence of a flat peak in the range 2θ = 25 ° indicates the presence of an amorphous structure [9][10]. The peaks 2θ = 42.5° and 2θ =52.2° in proportion to (4 0 0) and (4 2 2) confirm the presence of silicon in the sample [11]. Other peaks are related to the molybdenum substrate.

FTIR analysis of siliconized molybdenum sample is shown in Figure 7. Si-O-Si tensile vibrations are in the range of (1000-1250 cm^{-1}), Si-H tensile vibrations are in the range of (2000-2300cm^{-1}) and O-H vibration modes are in the range of (3000-3600cm^{-1}). The 1120 cm^{-1} and 1260 cm-1 peaks [11] correspond to Si-O vibration modes. As mentioned, peaks in the range of 2000-2300 cm-1 are related to Si-H .

The peak at 2282 cm^{-1} is in an oxygen medium. A silicon atom is bonded to one or more oxygen atoms and is defined as SiH (Ox) [12]. Si-O bonds indicates that the compound is oxidized in the air. The presence of a peak at (1450 cm^{-1}) is related to a weak Si bond. 1311 cm-1 and 1630 cm^{-1} are related to weak and medium Si-C bonds, respectively [13].

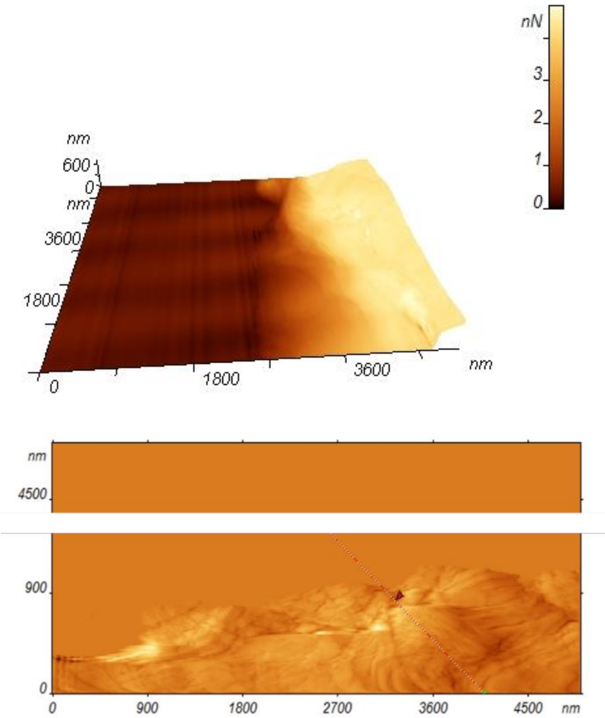

Fig. 4. The AFM image shows the thickness of the coated sample. Top) The thickness of the coating is determined by moving from left to right from the uncovered area to the covered area. Bottom) The surface of the coated sample with greater magnification.

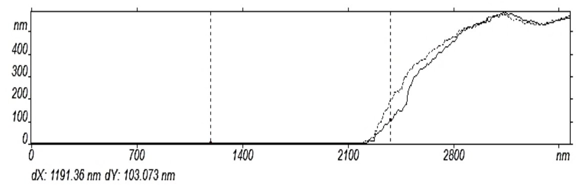

Fig. 5. Thickness of the coated area relative to the uncoated area in the molybdenum sample.

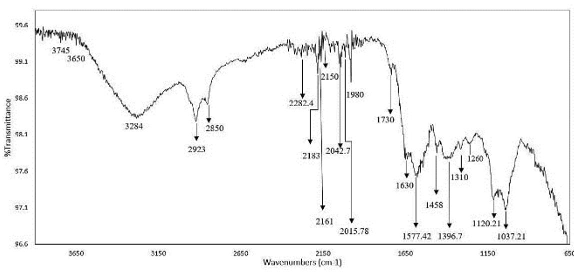

Fig.7. FTIR spectrum of siliconized molybdenum sample during 170 minutes.

4. Conclusions

In this work, siliconization of Mo sample using the glow discharge method has been successfully conducted using the glow discharge method with the gas mixture of 90%He+10%Si. The thickness of the coating has been estimated at around 450 nm using AFM. Xrd Analysis of the siliconized Mo sample indicates the presence of an amorphous structure.

EDX analysis of the siliconized Mo sample shows the formation of a silicon layer on the surface. The results showed that the glow discharge method could be used for siliconization of the tokamak diverter and limiter made of molybdenum.

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