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Metal oxide coating on first mirror in fusion reactor with carbon wall

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ABSTRACT

The lifetime of diagnostic equipment in a fusion reactor is typically very short. The first mirror used to reflect optical signals for diagnostics plays a crucial role in the reactor, and it is highly important to develop a more durable first mirror which can survive in the hostile environment. In this work, by conducting electron beam deposition on molybdenum substrates, metallic oxide mirrors are prepared and studied in the simulated environment. The multi-layered metal oxide mirror exhibits much higher reflectivity than the original molybdenum one and the *in situ* technique to monitor the performance of the first mirror is developed and described.

1. Introduction

As concerns about climate change intensify and fossil fuels will ultimately run out, nuclear power is a viable option for large-scale power generation especially in developing countries. Besides nuclear fission which is the technology adopted by nuclear power plants in the world, many experiments are being carried out to explore the fundamental and practical challenges facing nuclear fusion. The measurement systems constitute one of the hot research areas in fusion science and topics of intensive research include machine protection and plasma control. However, the development of the appropriate materials for the diagnostic systems is a big technical challenge because of the harsh environment in a fusion reactor as well as demanding measurement requirements. For instance, the materials must be able to withstand a high temperature, high level of radiation damage, large production rates of transmutation elements, and high thermo-mechanical stress. Furthermore, they are prone to deposition of materials eroded from the wall of the reactor. Hence, conventional diagnostic equipment cannot operate properly under these hostile conditions, including the first mirrors used to reflect signals for diagnostics, as shown in Fig. 1. The first mirrors are the first components for reflection and face the harshest environment. Dedicated programs have been initiated in a number of tokomaks to address the issue, but there is still no ideal solution to guarantee operational stability of the first mirrors [1].

First mirrors are installed in many locations in the fusion reactor with carbon walls, for instance, the vacuum vessel, diagnostic ports, interspace between the vacuum vessel and cryostat, outside the biological shield, and remote diagnostic areas. Those installed in the vacuum vessel close to the first carbon wall are subjected to the harshest environment due to the effects of chemical and physical sputtering. Previous studies have revealed the following main factors leading to degradation of the first mirror reflectivity: (1) deposition of impurities originating from in-vessel components exposed to the hostile environment and (2) erosion occurring on the first mirror due to charge exchange and neutral bombardment [2]. Generally, the two events occur simultaneously during the lifetime of the first mirrors. The conditions under which erosion prevails over deposition are often referred to as the net-erosion environment while the conditions are called the net-deposition environment if the deposition process is prominent. Mirrors which are located in the main vacuum chamber and have an open geometry towards the plasma are more likely subjected to net erosion, whereas mirrors located in the divertors and ducts with a closed geometry are in the net-deposition environment.

Molybdenum is preferred in the first mirror due to its low sputtering yield under deuterium exposure [3] and high hardness under pressure [4]. In addition, the reflectance of a crystalline mirror retains the initial value after long-term operation while that of a polycrystalline mirror tends to deteriorate rapidly under the same condition [5]. However, owing to the technological difficulties in producing a large single-crystal molybdenum mirror, a coating on polycrystalline molybdenum is an alternative solution. The objective of this study is to develop a promising and cost-effective first mirror that can operate under the harsh environment in a fusion reactor with a carbon wall. According to previous studies, obvious improvement in the reflectivity can be

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attained by a nanostructured rhodium coating [6,7], but Rh is quite expensive. It has also been reported that mirrors made of metal oxide have higher reflectance in the major diagnostic wavelength range between 800 and 1200 nm than ones composed of noble metals [8]. Herein, the properties of polycrystalline Mo mirrors coated with several types of multilayered structures such as Ta_2O_5, SiO_2, and ZrO_2 are investigated. In order to simulate the fusion reactor environment with a carbon wall and determine the behavior of our samples, carbon deposition, which is the predominant factor responsible for optical degradation of the first mirror close to the carbon wall in the fusion reactor [9], is investigated. The hardness of the first mirror is studied by nano-compression [10], and according to the dramatic change in the resistivity, an *in situ* technique to monitor the performance of the first mirror is developed and described.

2. Experimental details

The mirrors used in this study were composed of polycrystalline molybdenum. The Mo samples were cut into pieces 25 mm in diameter and 5 mm in length, polished with diamond paste up to 1 μm, cleaned in acetone and distilled water in an ultrasonic bath, and dried before deposition. In order to improve the reflectivity of the first mirror, electron beam deposition was conducted to deposit the Ta_2O_5, SiO_2, and ZrO_2 multilayered coatings on the Mo substrates. In order to cater to the primary wavelengths from 800 to 1,200 nm in fusion diagnostics, the thicknesses of the periodic Ta_2O_5 and SiO_2 multilayers [11] were 137 nm and 180 nm, respectively, to cover the range from 1,000 to 1,200 nm, and the thicknesses of the repeating ZrO_2 and SiO_2 multilayers [12] were 103 nm and 147 nm, respectively, for the 800 to 1,000 nm range. Besides, a transition layer consisting of 125 nm thick Ta_2O_5 was inserted between the two sets of periodical layers.

All the coatings were deposited on the AST PEVA-600E electron beam system. After the vacuum had reached 1×10^{-4} Pa at room temperature, the Mo samples were annealed at 350 °C for 1 hour *in situ*. The temperature of the substrates was measured by a thermocouple. The filament was biased to −10 kV to create the accelerating electric field and the ambient temperature was maintained at 350 °C during deposition. The metal oxide coatings were designated according to the number of layers as 1-layer, 2-layer, 5-layer, and 65-layer as shown in Fig. 2A–D.

As carbon deposition originates from chemical and physical sputtering of the carbon-containing first wall in a fusion reactor, the carbon contamination has relatively low energy and should be non-uniform. The first mirrors in the fusion reactors are vulnerable to the formation of non-uniform and reflected contamination coating, which increases the surface roughness and introduces deleterious effects to the optical properties [13,14]. In order to investigate low-energy and non-uniform carbon deposition, the simulated experiments were conducted by carbon paste deposition instead of sputter deposition. In carbon paste deposition, the carbon particles have low energy and form clusters, thus better resembling the actual situation in the reactor. The carbon paste was deposited from all directions on the samples at 1 Pa and 150 °C, both of which are typical parameters in a reactor [13,15].

In order to determine the thickness of the deposited layers, the 1-layer, 2-layer, and 5-layer samples were examined by ellipsometry on the J. A. WOOLLAM M-2000 [16] from 240 nm to 1,000 nm at 58° and 68°, respectively, while the 65-layer sample was observed by field-emission scanning electron microscopy (FE-SEM) on the HITACHI S-4800. All the samples, including original Mo mirrors and other metal oxide mirrors, were characterized by UV-VIS-IR spectrophotometry (SHIMADZU Solidspec-3700) in a wavelength range of 400 to 1600 nm to determine the initial and final reflectance [17]. Raman scattering was conducted on the HORIBA Scientific LabRAW HR Evolution and the spectra were deconvoluted by two Gaussian peaks to determine the chemical structure of the deposited carbon [18]. Nano-compression tests were performed to evaluate the hardness and displacement [19], and the surface electrical resistivity of the Mo mirrors and 65-layers was measured on the EVERBEING Wafer Probe Station with a KEITHLEY 4200-SCS [20].

3. Results and discussion

Ellipsometry is used to determine the thickness of the samples, and the results of the 1-layer, 2-layer, and 5-layer samples are shown in Fig. 2E–G. The ellipsometry parameters, Psi and delta, in the wavelength range of 240–1,000 nm are monitored at two incident angles of 58° and 68°, respectively, using a J. A. Woollam rotating polarization instrument [21]. The analysis is carried out by building a structural model for the different layers by the Woollam software. Beginning with a uniform thickness of the individual layer of 120 nm, the calculated data are fitted and the fitted data are listed in Fig. 2I. Taking into account minor deviations among samples, a coating with more periodical layers has less overall error in the individual thickness and enhances the reflectivity. The thickness of the 65-layer sample is determined by SEM (Fig. 2H), and the reflectivity is shown in Fig. 3A and C, which demonstrates that the 65-layer sample satisfies the individual layer demand and has the highest reflectivity among the samples.

The UV-VIS-IR results are displayed in Fig. 3A and C. Since the primary diagnostic wavelength is between 800 and 1,200 nm, our studies focus on this range. The calculated data before and after carbon contamination are shown in Fig. 3B and D for comparison. The calculated data in the range of 400–1,600 nm are obtained using Maxwell's [22] and diffuse reflection equations [23] as described below. According to Maxwell's equations, the *j*th film can be represented by the 2 × 2 matrix M_j as follows:

$$M_j = \begin{pmatrix} \cos\delta_j & \frac{i}{n_j}\sin\delta_j \\ in_j\sin\delta_j & \cos\delta_j \end{pmatrix}, \tag{1}$$

where $\delta_j = \frac{2\pi}{\lambda} n_j d_j \cos\theta_j$, λ is the light wavelength, θ_j is the angle of refraction, and n_j is the effective refractive index. The complete multilayer is represented by the product matrix M,

$$\mathrm{M} = \begin{pmatrix} m_{11} & im_{12} \\ im_{21} & m_{22} \end{pmatrix} = \prod_{j=1}^{k} M_j \tag{2}$$

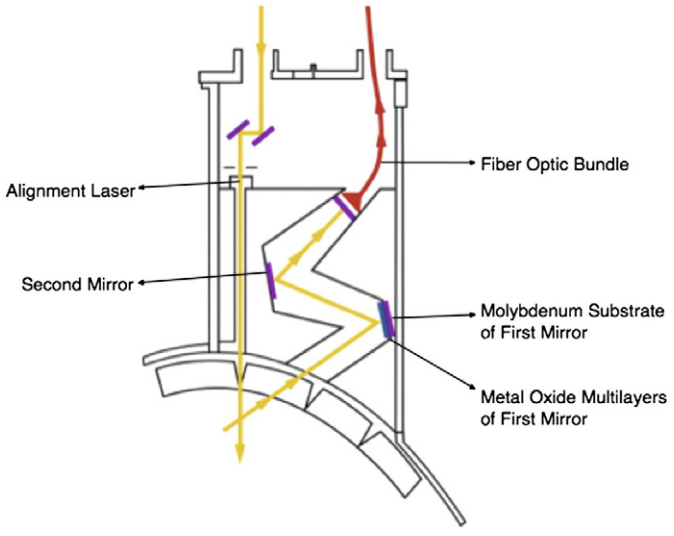

Fig. 1. Schematic showing the first mirrors in the fusion reactor.