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Development of a method to prepare thin metal films samples with controlled small aspect ratio roughness

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The surface topography is identified as one of the critical issues with regard to the processes of erosion and re-deposition in different fusion devices. In principle, the dependence of the sputtering yield with the angle of incidence for a rough surface deviates from that of a perfectly smooth surface because of the actual angle of incidence that the projectile sees on the rough surfaces. Besides that, the re-deposition phenomenon on a rough surface will further complicate the above scenario. Samples exposure of surfaces with average roughness > 0.1 μm (Ra) to ion beams have shown that for rough surfaces the dependence of the sputtering yield on the angle of incidence of beam particles is not as strong as for smooth surfaces. Understanding the sputtering and reflection dependence on the incident angle on such a rough surface is strongly advisable for the future fusion devices in order to know the impurities in the plasma scrape of layer when the W first wall is ageing. Although metal samples with Ra > 0.1 μm can be easily manufactured, thin film samples with roughness (reproducible and controlled) in the range of $0.01 < Ra < 0.1$ μm are rather difficult to obtain. In this work, we present a method to produce silicon-based metal samples with a very low controlled roughness by means of the formation of nano-scale features on the silicon (Si) substrate surface via plasma reactive ion etching (RIE). A tungsten (W) thin film (200 nm) is deposited by plasma sputtering onto the Si, reproducing the underneath texturing. A metallic titanium (Ti) interlayer (150-200 nm) has also been deposited between the substrate and the W film. Ti interlayer is likely to decrease the internal stress at the substrate–film interface, thus smoothing the difference in lattice parameters between the W and the Si. In this way, we are able to reduce voids and defects at the interface and to densify the top W coating.

Introduction

ITER will start operation with a combination of different materials for the plasma facing components (PFCs) including beryllium for the main chamber wall and tungsten for the divertor target, the region with the highest particle and heat loads [1].

Plasma/material interactions is the most critical engineering issue for fusion power development. Key concerns include the lifetime of PFCs due to steady state sputter erosion, plasma contamination by eroded material, tritium codeposition in redeposited material, and plasma operating limits due to these factors [2]. Regarding the erosion of materials, the physical sputtering which is the main cause of the erosion phenomena depends on multiple factors [3]. One of these is the surface roughness [4-5]. The surface roughness may influence the behavior of scattering of the incident ions too (reflection coefficient).

The effect of surface roughness on material sputtering has been already discussed, both in experiment and in modelling. In particular, sample exposures to ion beams have shown that for rough surfaces the dependence of the sputtering yield on the angle of incidence of beam particles is not as strong as for smooth surfaces [6-7]. Moreover, it was found that a significant fraction of sputtered atoms can be re-deposited on a rough surface due to geometry effects. This leads effectively to a decrease in the sputtering yield [8-9]. Regarding divertor materials, it was found that the erosion characteristics of tungsten, for example, in ASDEX-Upgrade [10], were affected by the surface topography. Different studies on berillium targets have found that the physical sputtering [11-12] decreases due to surface roughness. Nishijima et al [13] have shown that the sputtering yield of rough/fuzzy tungsten surfaces [14] under 110 eV Ar-ion sputtering decreases with increasing fuzz thickness and saturates at about 10% of that of a smooth surface.

In view of the above, it is necessary to investigate further the erosion of W to understand the relations between ion interaction and surface roughness.

In order to provide W specimens to be analyzed with surface science technologies (ion beam analysis), in this work we report on a Reactive Ion Etching (RIE) [15] plasma process able to produce thin metal W coatings on a silicon substrate with the desired features. In particular, we produce specimens that show a W surface with a very low controlled small aspect ratio roughness. A metallic Ti interlayer was also deposited at the interface between Si and W in order to decrease the internal stress [16] and produce a dense and defect free coating. The produced samples were provided to all partners within the PFC.SP2.4 EUROFUSION activity to measure the erosion yield as a function of ion incidence angle.

Experimental Details

The plasma reactor

An RF plasma system [17] has been used to produce a physical structuring of Silicon (type P, dopant B, <100>, 0.01-0.02 Ohm-cm, 1 × 1 cm^2, thickness = 400 μm). The experimental apparatus consists of a parallel-plate, capacitive-coupled system, made up of a cylindrical stainless steel vacuum chamber with an asymmetric electrode configuration. A powered electrode (3-in diameter) is connected to an RF (13.56 MHz) power supply, coupled with an automatic impedance matching unit, while the other electrode (3-in diameter), made up of stainless steel, is grounded. Si substrates are placed on the powered electrode at 6 cm away from the ground electrode. Before the process, the substrates are cleaned with alcohol followed by rinse in deionized water to remove surface contaminants. The Atomic Force Microscopy technique has been used to check the surface roughness of substrates after cleaning. The RMS roughness was in accordance to manufacturer's data (≤ 1 nm). The process chamber is pumped to a base pressure below 1×10^{-5} Pa and high-purity CF_4 and H_2 gases (10% H_2) are introduced into the vacuum chamber through a mass flow controller in order achieve the desired working pressure, which is fixed at 9 Pa. The plasma process was performed for 20 min with a RF power of 250 W.

After textured process of Si, before W deposition, a Ti interlayer was deposited by diode sputtering of Ti target (3-in diameter, purity 99.9%). Textured Si was placed onto the grounded substrate holder kept at 4.5 cm distance from the RF powered electrode. The vacuum before deposition was less than 1×10^{-5} Pa and high purity Ar (99.995%) gas was introduced into the chamber through a mass flow controller and a gate valve was used to adjust the pressure at 3 Pa during the process. A radiofrequency power source, capacitively coupled with the deposition chamber, was fixed at 100 W obtaining a deposition rate of 10 nm/min. After the Ti interlayer deposition, a W coating was deposited by diode sputtering of W target (3-in diameter, purity 99.995%) using high purity Ar (99.995%) at a pressure of 0.8 Pa and 100 W of RF power. In this case the deposition rate was 3.3 nm/min.

Characterization of the nanostructured surface

The morphology of nanostructured surfaces was examined by High-resolution Scanning Electron Microscopy (SEM) imaging.

Atomic Force Microscopy (AFM) measurements were made in air by a Nano-RTM AFM System (Pacific Nanotechnology, Santa Clara, CA, USA) operating in close contact mode. Silicon conical tips of 10 nm radius mounted on silicon cantilevers of 125 m length, 42 N/m force constant and 320 kHz resonance frequency were used. Images were processed and analyzed by means of Gwyddion, an open-source software for AFM data analysis [18].

Results and Discussion

In order to produce a W top-coating with a rough surface with a specific and controlled aspect ratio, a plasma etching process [15] with a subsequent process of sputtering deposition was developed and applied on Si substrate (1.5 x 1.5 cm^2).

The plasma etching of Si was produced by means of the fluorocarbon and hydrogen molecules used during the process. Under suitable plasma conditions [19-20], the molecules undergo fragmentation and ionization, and the main species produced are CF_x ,F and H radicals (CFx*, F*and H*) and CFx^+ ions. When F* radicals react with Si, a volatile compound SiF_4 is formed, resulting in the surface etching. In addition to this chemical etching a thin passivating fluorocarbon (nCF_2) film is deposited randomly onto the Si surface, obtaining an auto masking effect towards the etching by F*. However the passivation layer is partly removed by the simultaneous CF_x^+ ions bombardment. The competition between passivation layer deposition and its removal by energetic ions bombardment cause local variation of etching rate on the Si surface, leading to the growth of random silicon nanostructures over the process time. The surface morphology can be adjusted by changing the RIE parameters, such as gas mixture composition, flow rate, system temperature, reaction time, substrate bias and RF power [15].

In Fig.1 are shown the SEM images of the Si textured after the plasma process.

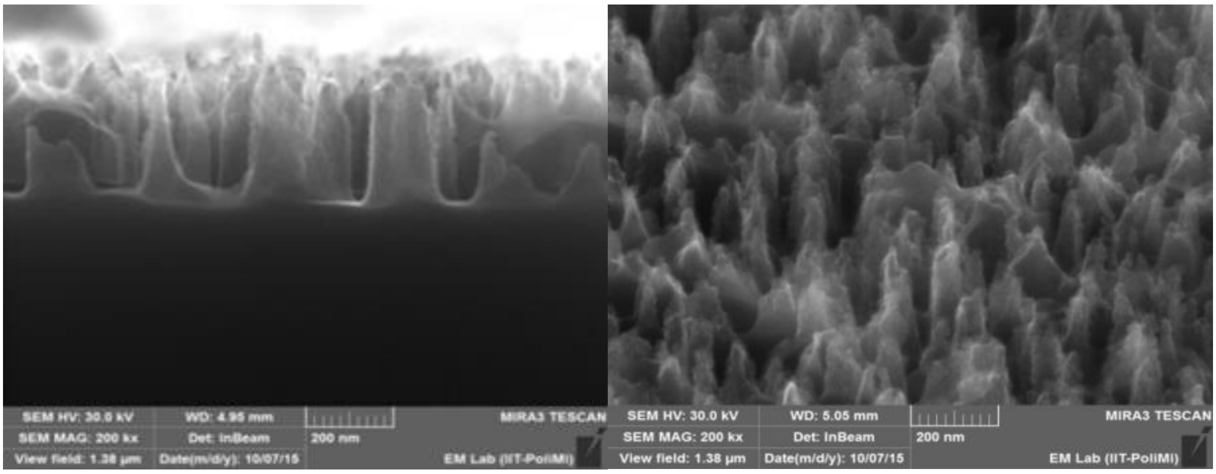

Fig.1 SEM Cross section image (left) and tilted image (right) of the textured silicon surface, taken at 30keV of Primary Electron energy. (SEM micrographs from Lab. CNR-IFN, Tescan MIRA III)

After the substrate texturization, a deposition of a Ti interlayer was necessary in order to produce a dense and uniform W coating. In fact as shown in Fig. 2, the direct deposition of the W onto the textured silicon lead to the formation of defects and voids at their interface. Moreover the W top coating has a morphology with well-defined and separated columnar structures.

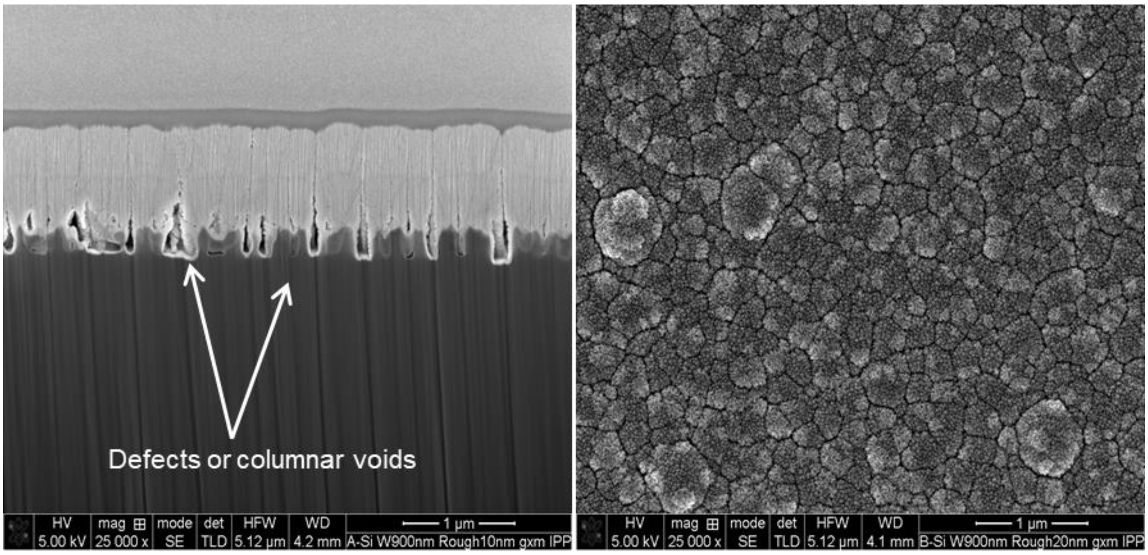

Fig.2 SEM Cross section (left) and top (right) images of the tungsten coating deposited directly onto the silicon substrate. (SEM micrographs from Lab. IPP Garching)

The deposition of a Ti interlayer is a standard practice used to improve the adhesion and the stability of different coatings obtained by physical vapour deposition (PVD). This effect is belived to arised from the combination of two factors.: the dissolution of weak native oxide layers by the titanium (a chemical gettering effect) and the fact that the interlayer behaves like a mechanical buffer. In fact, the titanium creates a soft interlayer, which reduces the shear stresses across the coating-substrate interface and prevents the propagation of cracks, inducing the growth of a dense and continue coating [16, 21]. Different interlayer thicknesses were deposited onto the substrate in order to find the optimal one in terms of the W coating uniformity. A Ti interlayer thickness of 150-200 nm (Fig. 3) turned out to be the most suitable for this purpose.

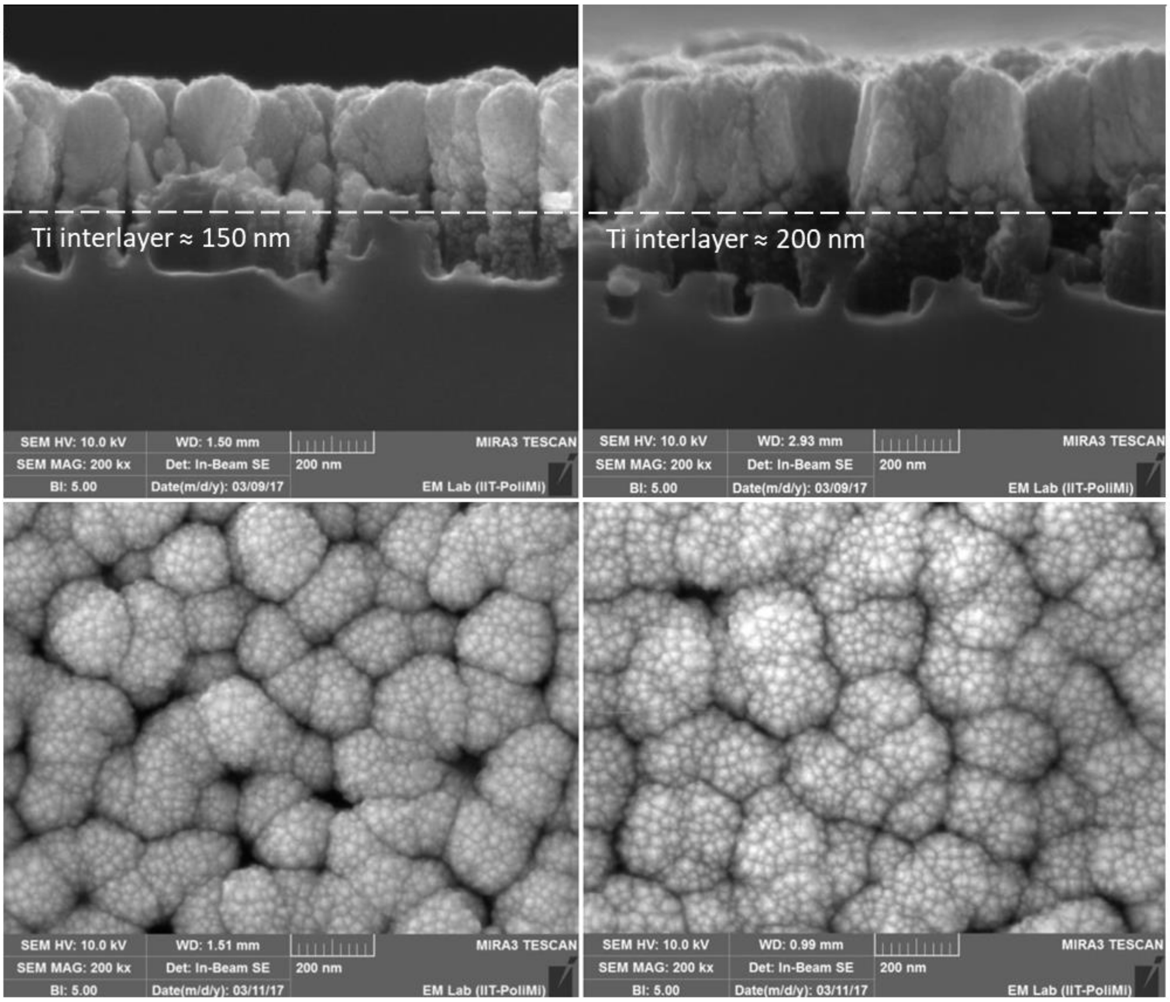

Fig.3 SEM Cross section and top images of the tungsten coating deposited onto the silicon substrate with titanium interlayers of 150 nm (left) and 200 nm (right). (SEM micrographs from Lab CNR-IFN, Tescan MIRA III).

In both cases, there is a clear densification of the top W coating and the defects at the interfaces are reduced compared to the sample without the Ti interlayer (Fig. 2). Moreover from these images is also possible to notice the effect of the interlayer thickness. In fact, in the sample with 150 nm of Ti interlayer is still possible to distinguish the separated columnar structures of the W coatings. Instead, by increasing the interlayer thickness by 50 nm, the columnar structures tend to merge, leading to a more dense and uniform coating. AFM images of the nanostructured W coating with a Ti interlayer of 200 nm were also performed and reported in Fig. 4. From these, it is possible to appreciate how the nanostructuring is continuous and homogeneous on the whole surface .The average roughness is about 10 nm and the average height value of the surface nanostructures is around 50 nm.

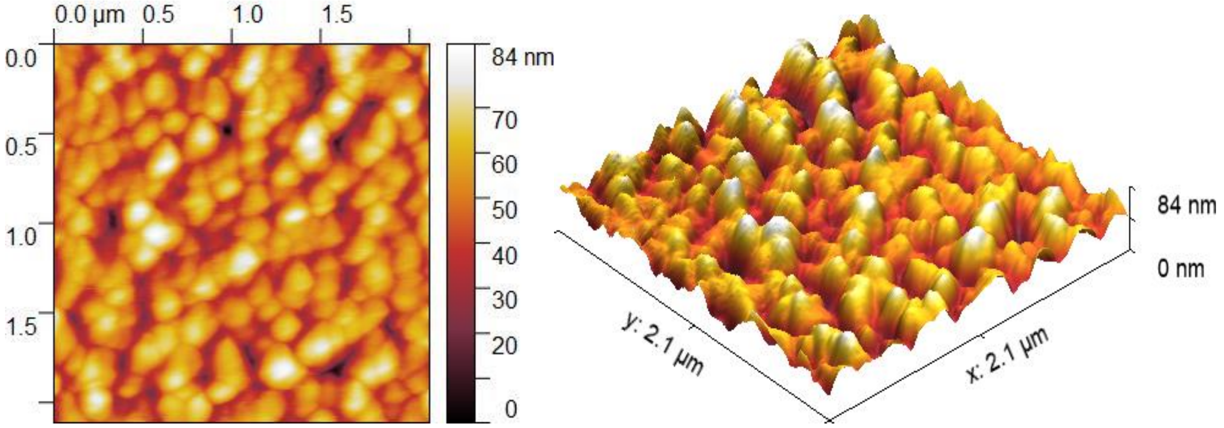

Fig.4 AFM image (left) and 3D reconstruction (right) of the nanostructured tungsten coating.

Conclusion

Metal-based samples with very low and controlled roughness were successfully obtained, by depositing a thin film of W by plasma sputtering on a Si substrate previoulsy textured by a RIE process. A deposition of a Ti interlayer was necessary in order to reduce voids and defects at the interface Si/W and to densify the W top coating.

This method can be used to to produce W specimens in order to study the effect of low surface roughness (≈10 nm) on the sputtering yield. Moreover the RIE process could be also exploited to nanostructure directly surface of bulk metals. Different gas mixtures must be used depending on the materials to be treated in order to obtain local variation of the etching rate of the surface and thus the nanostructurization [22-23].

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