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Near surface modification of R-sapphire exposed to long term RF hydrogen-neon plasma

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Samples of R-sapphire were exposed to long term RF hydrogen-neon plasma. Modification of near-surface layers was studied by XRD and XPS, and optical changes were assessed by spectrophotometry. After removing 270 nm thick layer the internal stresses remained at an initial value, but in the layer that probing by XRD individual blocks turned around and shifted from the Bragg reflection. XPS shows that aluminum is still an Al_2O_3 compound. Light transmission in the wavelength range 400−1000 nm remained unchanged which makes it possible to use sapphire as a protection window for optical diagnostics in fusion facilities.

Keywords: sapphire, RF discharge, hydrogen, neon, structure surface layer, light transmission, ITER.

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Several optical diagnostics, which include the diagnostics of Thomson scattering (DTS) of a laser beam by plasma electrons [1], will be used to analyze plasma parameters in different modes at the International Thermonuclear Experimental Reactor (ITER). In DTS, the probing laser beam is scattered by plasma electrons and collected by the so-called „primary" mirror (made, e.g., of molybdenum) that faces the plasma. The primary mirror is subjected to significant thermal loads and the influence of neutral atom fluxes from plasma [2]. To preserve its optical properties, one needs to develop a system of protection against the adverse effects of plasma and impurity atom fluxes. Films of Be or B [3] (in the modified ITER design) deposited onto the mirror will reduce its reflectance. It was proposed in [4] to use a radio-frequency (RF) gas discharge to clean the primary mirror. The possibility of protecting the primary DTS mirror with a sapphire window was considered in [1]. The protective window will require cleaning in this case.

Sapphire (α-Al_2O_3) is a chemically inert, durable, and heat-resistant dielectric with high thermal conductivity, which is transparent in the wavelength range of most optical diagnostics (300−1100 nm) [5]. In the sapphire lattice, Al vacancies are positioned on the so-called r-planes with Miller indices $(1\bar{1}02)$, making sapphire cleavable along these planes. The r-plane in our experiments was parallel to the surface of wafers, which are hereinafter referred to as r-C sapphire wafers.

Relying on the results of experiments on tokamak wall conditioning [6], we chose a mixture of neon and hydrogen (77%H_2−23%Ne) was chosen as the cleaning gas for an RF discharge. In preliminary experiments, a 30-nm-thick Al film was deposited onto the r-C surface three times, and the wafer was cleaned in an RF discharge for 1 h after each deposition. The light transmittance within the 400−1000 nm wavelength range did not change after three deposition and cleaning cycles.

Owing to the non-uniformity of removal of contaminants from the r-C surface in the process of cleaning, the surface layer of the sapphire window will be exposed directly to plasma for a certain period of time. Therefore, for the application of an RF discharge to be efficient and reliable, one needs to monitor the changes in structure and topography of a clean optical surface in the course of a sufficiently long plasma exposure and the associated change in light transmittance of r-C.

The influence of long-term (12 h) exposure to a cleaning RF discharge on the structure and composition of the surface r-C layer and on the optical stability of sapphire (in terms of its light transmittance) is analyzed below. The light transmittance within the operating wavelength range of DTS (400−1000 nm) was chosen as the primary criterion of optical durability of the window material.

Square sapphire wafers ($10 \times 10 \times 1$ mm) polished optically on both sides were used in experiments. The setup and the analysis techniques were discussed in detail in [7,8]. The pressure in the mixture and the bias at the sample were kept constant throughout the discharge process: 15 Pa and −300 V. The sample temperature did not exceed 100°C. With this pressure, the energy of ions and neutrals incident onto the sample varies from a near-thermal one to 300 eV. According to the results of calculations in SRIM [9], the maximum range of 10 nm corresponds to atomic hydrogen ions H^+. The addition of 23% of neon to hydrogen plasma increased the r-C sputtering rate from 8.9 to 22.5 nm/h and was not accompanied by the introduction of impurities into the surface layer examined by energy-

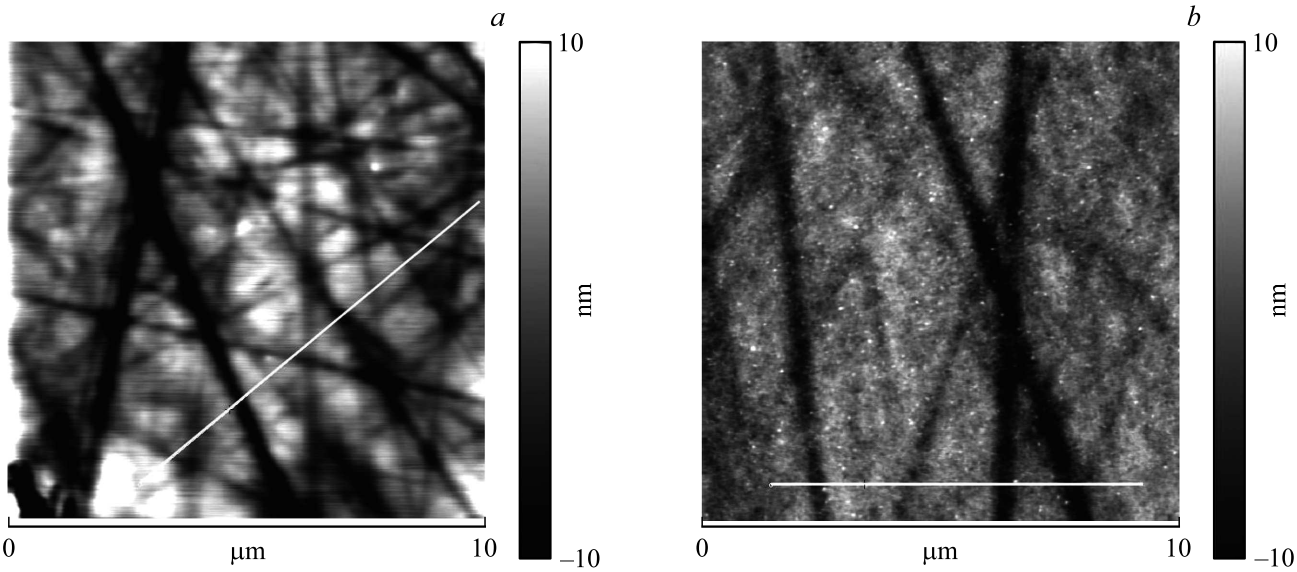

Figure 1. AFM images of the r-C ($10 \times 10\,\mu$m) surface after mechanical polishing (a) and exposure to plasma (b).

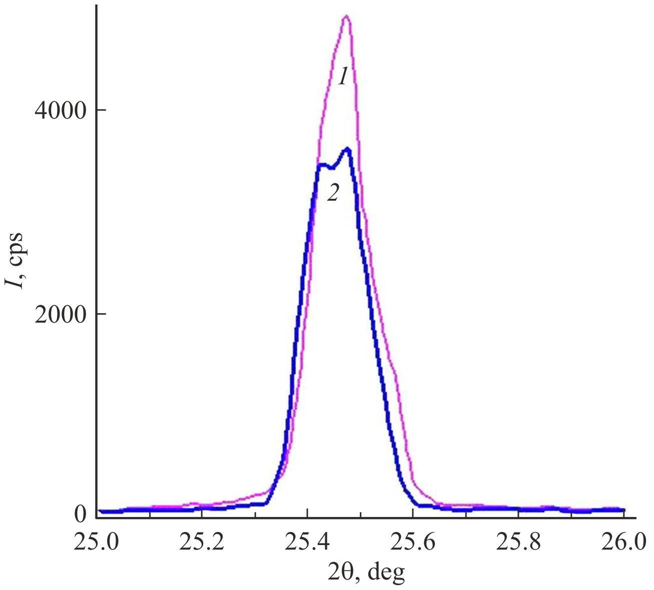

Figure 2. XRD spectra of the r-C wafer measured before (*1*) and after (*2*) exposure to an RF discharge and averaged over azimuthal angle φ ($\theta-2\theta$; CuK_α radiation; the sample was rotated about an axis normal to the surface).

dispersive spectroscopy. The thickness of the sputtered layer subjected to an RF discharge was 270 nm.

According to the atomic force microscopy (AFM) data, polishing grooves with a depth up to 8–10 nm were the main defects on the original wafer surface (Fig. 1, a). The root-mean-square roughness was $R_q = 5.7$ nm. Polishing grooves with a depth up to 10 nm remained visible after the exposure to an RF discharge (Fig. 1, b). The analysis of scratch profiles performed along the white lines in Fig. 1 revealed the presence of protrusions or banks as high as 10 nm along the scratches on the original wafer. Following a discharge, the height of these banks decreased to 4–6 nm, while the roughness dropped to $R_q = 3.7$ nm.

The presented data are indicative of gradual removal of grooves, which is attributable to lowering of the sputtering rate on the bottom of scratches relative to the corresponding rate for the flat surface and the banks rising above it.

According to the X-ray reflection diffraction (XRD) data, the key change in the spectrum induced by plasma processing is a reduction in the intensity of reflection $(1\bar{1}02)$ compared to the intensity of this reflection in the original wafer (Fig. 2). The weakening of this reflection in the irradiated wafer suggests a reduction in the number of blocks forming the Bragg reflection. Two maxima at angles $2\theta = 25.42$ and 25.48° formed in reflection $(1\bar{1}02)$ with interplanar spacing $d = 0.3490 \pm 0.0002$ nm. The emergence of two maxima is associated with a 0.06° rotation of blocks tens of micrometers in size. The slight skew of the $(1\bar{1}02)$ reflection shape on the left side of the wafer observed prior to exposure to an RF discharge makes it is evident that this rotation was already emergent in the original wafer. Plasma processing has just triggered its completion.

Since the linear coefficient of absorption of Cu- radiation in r-C is $\alpha = 125\,\text{cm}^{-1}$ [8], a crystal layer with a thickness up to $40\,\mu$m is involved in the formation of a Bragg reflection from r-planes. Only a fraction of blocks in this layer are in the exact ($\pm 0.04^\circ$) Bragg position during symmetrical measurements. The weakening of reflection from the r-planes is probably caused by rotations of individual blocks with a size of several micrometers by angles greater than 0.1° as a result of sputtering of a 270-nm-thick layer. These rotations may be induced by significant internal stresses present in the original wafers.

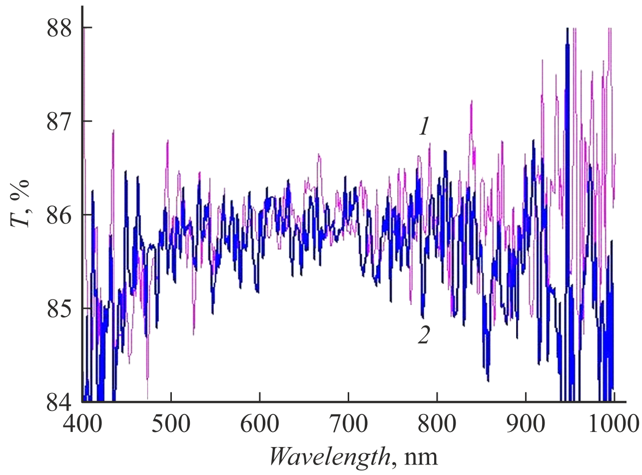

Figure 3. Light transmittance (T) of r-C wafers before (*1*) and after (*2*) 12 h of plasma processing.

The initial level of light transmittance within the 400–1000 nm wavelength range remained virtually unchanged after 12 h of exposure in an RF discharge (Fig. 3).

According to the X-ray photoelectron spectroscopy data, aluminum in the surface layer with a thickness of approximately 2 nm was in an oxidized trivalent state.

It was noted in literature [10] that selective sputtering should be enhanced near the oxygen sputtering threshold [11]. This is attributed to the preferred transfer of energy from hydrogen ions to the light component of the target. However, the contributions of fluxes of impurity atoms and molecules of oxygen and neon to the total sputtering coefficient may neutralize partially the effect of sapphire reduction. Selective sputtering and enrichment of the surface layer with aluminum were not detected in the discussed experiments.

The obtained data revealed that surface roughness R_q decreased from 5.7 to 3.7 nm after exposure to an RF discharge and removal of a 270-nm-thick material layer. Plasma processing had a polishing effect on the original surface with grooves. Rotations of individual blocks and their shift from the Bragg position were observed in a layer with a thickness up to 40 μm in r-C wafers with significant initial internal stresses subjected to an RF discharge.

The stoichiometric composition of both the uppermost nanometer-thick layers and the deeper micrometer layers has remained unchanged. Selective sputtering of oxygen in a reducing environment with hydrogen atoms and ions was not observed. The transmittance did not undergo any noticeable changes within the studied wavelength range.

The demonstrated stability of light transmittance of r-C wafers subjected to an RF discharge in a 77%H_2–23%Ne mixture suggests that the considered method of surface cleaning holds promise for restoration of the light transmittance of protective windows in the DTS in front of the primary mirror at the ITER tokamak.

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Conflict of interest

The authors declare that they have no conflict of interest.

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