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# BEHAVIOR OF METALLIC DIAGNOSTIC MIRRORS WITH DIFFERENT STRUCTURES UNDER CONDITIONS SIMULATING THOSE IN THE ITER FUSION REACTOR

*In the paper by V.S. Voitsenya et al. (Plasma Phys. Rep.* ***20****, 217 (1994)), a methodology aimed at an optimal selection of materials for in-vessel mirrors used in optical and laser methods of plasma diagnostics in the experimental fusion reactor ITER was elaborated. The corresponding systematic simulation studies concerning the behavior of mirror specimens fabricated from different metals with different structures – polycrystalline (Be, Al, SS, Cu, Ti, Mo, W, Ta), single-crystalline (SS, Ni, Mo, W), and film (i.e. the film/substrate structure, namely, Be/Cu, Cu/Cu, Rh/Cu, Rh/V, Rh/SS, Mo/SS, Mo/Mo) – as well as mirrors fabricated from amorphous alloys of the ZrTiCuNiBe type, under long-term sputtering by deuterium (in some cases, argon) plasma ions were carried out. Amorphous mirror specimens were shown to be much more resistant to the development of roughness in comparison with mirrors with any other structure, which results from the complete absence of any ordered structure on the surface on a scale exceeding a few nanometers. The most important results were confirmed experimentally on such fusion installations as the TEXTOR (Jülich, Germany), ASDEX-U (Garching, Germany), and Tore Supra (Cadarache, France) tokamaks, the heliotron Large Helical Device (Toki, Japan), on the small tokamak TRIAM-1M (Kyoto, Japan), and on special stands at Lausanne University (Switzerland) and in the Institut für Plasmaphysik, Association EURATOM-FZJ, FZ-Jülich (Germany).*

*Keywords*: plasma, ITER, diagnostic mirror, surface ion bombardment, relief, metallic mirror structure, mechanism of relief formation.

## 1. Introduction

The experimental thermonuclear reactor ITER is under construction in France. Formally, 34 states participate in this project. The "first plasma" is planned to be generated in 2020. Hydrogen will be used as a working gas at the first stage of the ITER exploitation. Nevertheless, all systems of the reactor diagnostics and control are developed with regard for real operational conditions, when the reactor will work on a deuterium–tritium mixture and all in-vessel elements of diagnostic complex will undergo rather high fluxes of neutrons and $\gamma$-radiation.

When the experimental thermonuclear reactor ITER works in a certain selected regime, a lot of various plasma parameters should be monitored in detail both in the plasma confinement volume and a divertor. Approximately 45% of all plasma parameter measurements will be carried out with the help of optical and laser diagnostic methods in a wide range of electromagnetic waves. Owing to high fluxes of $\gamma$ and neutron radiation from thermonuclear plasma, all optical measurement schemes have to be based on metallic mirrors, because the optical properties of dielectric elements (lens, prisms) quickly deteriorate under those conditions. Therefore, every optical scheme will include a mirror (the so-called first mirror, FM), which faces the plasma. Its location is closest to plasma in comparison with any other element of the scheme such as a second mirror (or a number of such mirrors), lenses or fiber optic light guides mounted in a periscopic labyrinth, and an output window, as is schematically shown in Fig. 1. All those elements will undergo, to different extents, the action of radiation from thermonuclear plasma, but the severest operational conditions will be realized for the first mirror. Besides $\gamma$ and neutron radiation, the latter will be subjected to the action of electromagnetic radiation in the interval from soft x-ray to infra-red wavelengths, as well as to the bombardment by charge exchange atoms (CEAs) with a wide energy spectrum (with an average energy of several hundreds of electron-volts [1]) and approximately with the same flux density as the flux density of CEAs on the first reactor wall, i.e. $\approx 2{\times}10^{15}$ atom/cm$^2$/s. The influence of electromagnetic radiation can be minimized by introducing an additional cooling of the mirror. At the same time, neutrons and, especially, CEAs will change the optical properties of mirrors: the former owing to the accumulation of various defects in the mirror material, and the latter owing to the sputtering of the mirror surface, which will give rise to its roughness and, as a result, the degradation of optical properties. The cumulative effect of both those factors may bring about the appearance of new synergetic phenomena, which should be analyzed beforehand in experiments that simulate the behavior of mirrors in the ITER. Within the reactor exploitation time interval, the thickness of the sputtered layer on the first mirror will reach a value ranging from several microns (for tungsten) to several tens of microns (for molybdenum, rhodium, and copper), which will induce the appearance of roughness and give rise to the degradation of optical properties.

In this review, the following basic results are presented and discussed:

• the results of simulation researches devoted to the behavior of metallic mirrors with polycrystalline (Al, stainless steel (SS), Cu, Ti, Mo, W, Ta), single-crystalline (SS, Ni, Mo, W), and film (film/substrate Be/Cu, Cu/Cu, Rh/Cu, Rh/V, Rh/SS, Mo/SS, Mo/Mo) structures, as well as mirrors fabricated from amorphous alloys, at their long-term sputtering by deuterium plasma ions;

• the results of simulation experiments concerning the simultaneous influence of neutron irradiation and charge exchange ions on metallic mirrors, the latter being promising to be used as in-vessel mirrors (Cu, SS, W) for plasma diagnostics in the ITER;

• the model describing the development of microrelief roughness on the surface of polycrystalline mirror under its sputtering;

• the chemical processes in the hydrogen plasma with carbon and oxygen impurities.

## 2. Influence of Deuterium Plasma on the Behavior of Metallic Mirrors with Various Structures

### *2.1. Polycrystalline mirrors*

Experiments with polycrystalline specimens of mirrors fabricated from Be, Al, SS, Cu, Ti, Mo, W, and Ta showed that it is impossible to find such metals among those with the polycrystalline structure that would preserve their reflecting properties to be constant at the long-term sputtering. When the surface of polycrystalline mirror is sputtered, a relief is developed, because differently oriented grains of metal are sputtered at different rates. In such a manner, there emerges a step-like structure on the surface [2, 3] connected with the orientation of grain faces. One can easily see this phenomenon on a micrograph (Fig. 2), where the Electron Backscatter Diffraction (EBSD) method was used to simultaneously register the face orientations of all grains on the surfaces in the microscopic field [4]. In addition, a relief with an even finer structure develops on the surface of some grains, with the probability of its appearance depending on the grain orientation, as was described, e.g., in work [4]. In the case of stainless steel, the facet (111) turned out the most stable with respect to the relief development, being also characterized by the largest sputtering coefficient.

Similar experiments were carried out for mirrors fabricated from polycrystalline tungsten [5]. Qualitatively, their results coincide with the data obtained for SS mirrors, if we take into consideration that stainless steel and tungsten are fcc and bcc, respec-

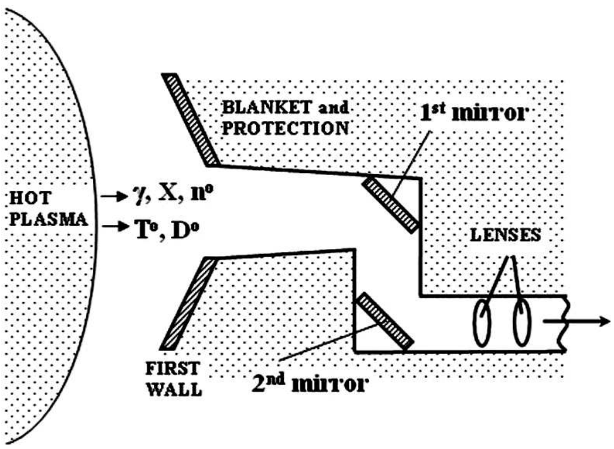



***Fig. 1.*** Experimental setup for optical measurements

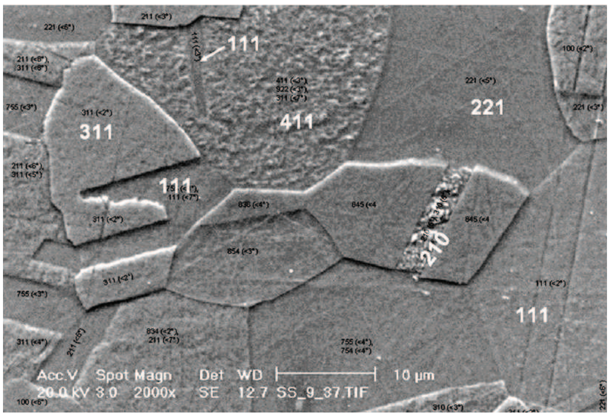



***Fig. 2.*** Surface relief of a mirror fabricated from stainless steel (SS) after irradiation with hydrogen ions. Figures on the grains indicate the orientation of the corresponding surface. One can see that grains with orientation (111) or close to it are sputtered more strongly, but have a smoother surface in comparison with grains with other orientations

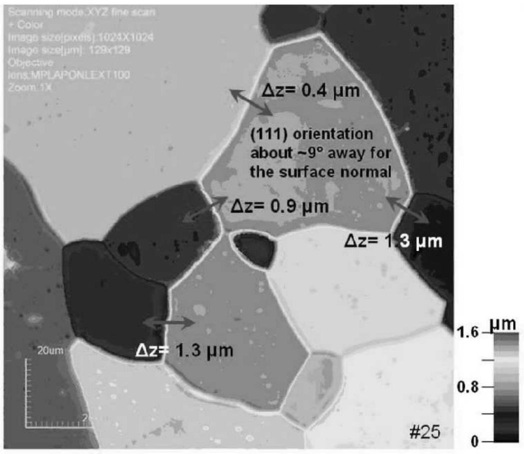
tively, metals. In other words, the facet (111) of tungsten, in contrast to steel, turned out the most stable with respect to the sputtering, as is seen from Fig. 3, whereas the grains with the edge orientation (110) were found to be the most sputtered. One can also see that an insignificant deflection of the face ($\approx 9^\circ$) from the exact orientation [111] did not change its priority in comparison with other facets.

The changes in the surface morphology mentioned above worsen the optical properties of mirrors. The surface roughness and, accordingly, the degradation rate of the mirror reflectance $R$, as was found in works [2,3] for the first time, grow, as the energy of ions that sputter the mirror surface increases. Figure 4, *a* illustrates the dependence of $R$ for SS mirrors at the normal incidence of light (a wavelength of 600 nm) on the thickness of a layer sputtered by hydrogen plasma ions with various energies, and Fig. 4, *b* the dependence of $R$ on the ion energy for a sputtered layer thickness of 4 $\mu$m (the thickness was determined from the loss of specimen mass). As is seen from scanning electron microscopy data [3], such a behavior of $R$ stems from a higher surface roughness developing in the case of a higher energy of particles bombarding the surface if the latter is sputtered to the same average depth. This fact can be connected only with the circumstance that the difference between the sputtering rates of metal grains with different orientations grows with the energy of bombarding ions. This result has almost not been discussed in the scientific literature earlier.

In work [6], the results of long-term (for several years) experiments with polycrystalline metal mirrors characterized by various crystal dimensions – from tens of nanometers to tens of micrometers – were analyzed. In particular, mirrors fabricated from the amorphous metallic alloy (AMA); mirrors obtained by annealing the AMA (a crystallite size of 30–70 nm); film mirrors (an Rh film on the Cu substrate, a crystallite size of 100 nm); mirrors from fine crystalline Cu, Mo, and W (a crystallite size of 250–350 nm); mirrors from the ITER-grade tungsten (the crystal size equaled 1–3 $\mu$m along the surface and about 5 $\mu$m perpendicularly to it), and specimens of tungsten mirrors recrystallized at a temperature of 2073 K after the polishing (a grain size of 10–100 $\mu$m [5]) were considered. The data on the relief obtained with the help of an atomic force microscope or laser profilometer were treated mathematically. On the basis of the relevant analysis, a model of the roughness evolution, when a mirror with the polycrystalline structure is sputtered, was developed (see its detailed description in work [6]).

The model is based on the known fact that the sputtering coefficient of separate grains in a polycrystalline metal depends on the orientation of the grain crystallographic axes with respect to the surface. It is reasonable to suppose that an insignificant deflection from the exact orientation of crystallographic axes, e.g., within the limits of $\pm 15^\circ$, is not followed by any appreciable variations in the sputtering coefficient. This assumption was qualitatively confirmed, while comparing the parameters of a relief appeared on the surface of polycrystalline W mirror specimen after its long-term sputtering and the measurement results of the orientations of faces of all grains on the surface using the EBSD method. The matter is that, if the facets with the orientations close to (111) deflect by about 9° from the true position (Fig. 3), they are all the same located well above the facets with other orientations [5].

The idea of the model is as follows. If no special means of the metal texturing are used – e.g., those which are applied when single crystals are grown up – the probabilities of the grain orientation in a metal at its cooling are identical for all directions from 0 to 360°. Therefore, there exists a high probability that a number of grains with slightly different orientations

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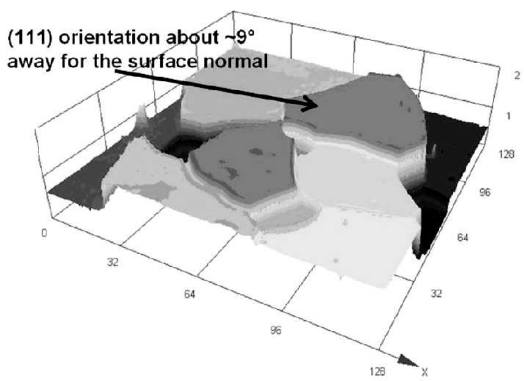


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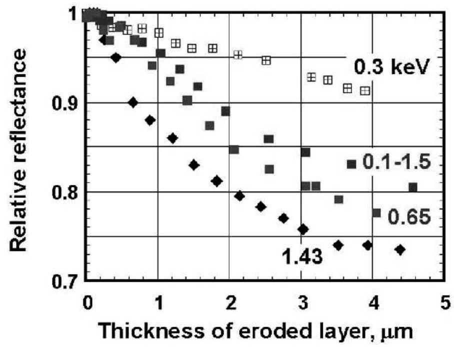
***Fig. 3.*** Cumulative EBSD and laser profilometry data. Two- (*a*) and three-dimensional (*b*) images of the surface of a polycrystalline tungsten mirror after its 4-$\mu$m (on the average, determined from the mass loss) sputtering with 600-eV argon ions [5]. The highest grains deflect by about 9° from the normal position of facet (111)

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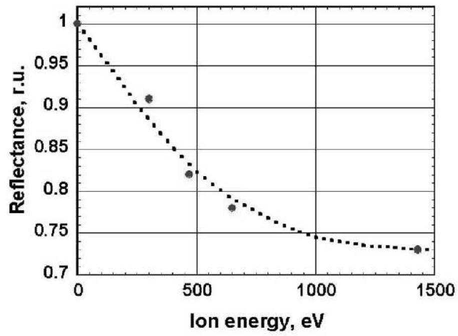


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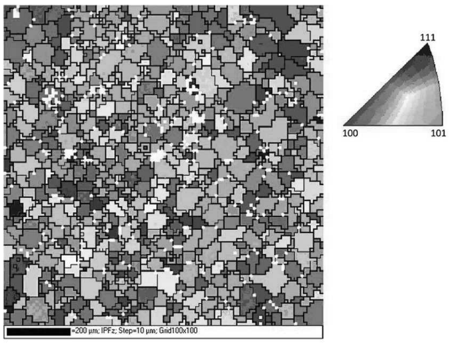
***Fig. 4.*** Reflectance degradation of mirrors fabricated from stainless steel at their irradiation with hydrogen plasma ions of various energies. The average energy of ions (470 eV) with a wide energy distribution was found, by taking into account that there are simultaneously ions $H^+$, $H_2^+$, and $H_3^+$ in hydrogen plasma [3]

will form a more or less isolated group somewhere on the surface, and the grains in this group will have approximately the same sputtering coefficient; at the same time, the grains with a different average orientation will form another group at a certain distance, and the latter group will be characterized by a substantially different sputtering coefficient. In other words, a relief with the longitudinal dimension characterized not only by the sizes of separate grains but by the dimensions of such groups and the distance between them will start to develop on the surface.

Figure 5, *a* obtained using the EBSD method while analyzing the surface of polycrystalline tungsten specimen (a scaled up section of this micrograph is shown in Fig. 3) is a good illustration of the appearance of groups with close orientations of grains within the group, but with different orientations between the groups. The stereographic triangle indicates the orientations of separate grains, and one can see that there are a lot of grain groups with a small color differences within every group, i.e. with a small difference between the edge orientations and, accordingly, the sputtering coefficients.

The described model was applied to treat the relief data obtained with the help of an atomic force microscope for the mirror specimens indicated above, except for the crystallized amorphous (here, the surface was examined on a scanning electron microscope) and recrystallized tungsten (here, the data of laser profilometry were analyzed) ones. The distribution functions for longitudinal surface roughness nonuni-

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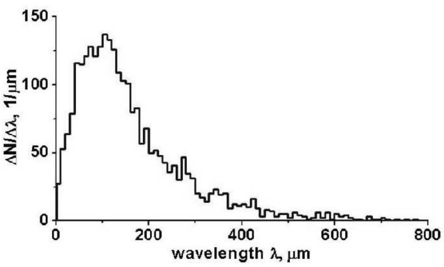


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***Fig. 5.*** (*a*) EBSD results obtained for a polycrystalline tungsten mirror after its sputtering by 4 $\mu$m; (*b*) profilometry results for the size distribution of relief inhomogeneities over the surface of this mirror specimen

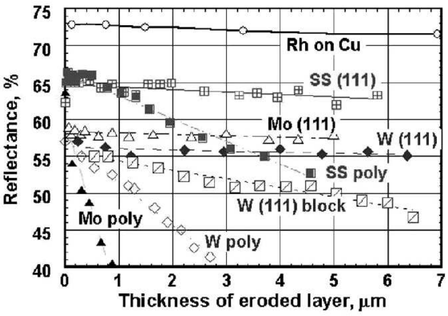


***Fig. 6.*** Reflectance dependences on the sputtered layer thickness for mirrors fabricated of some polycrystalline and single-crystalline metals and a mirror with a Rh film on the Cu substrate. The notation "W(111) block" marks the data obtained at the sputtering of polycrystalline texturized tungsten with the orientation (111) for the majority (about 96%) of its grains

formities were obtained. A typical example of the W mirror specimens is depicted in Fig. 5, *b*. One can see that the spectrum contains nonuniformities with dimensions along the surface much longer than the dimensions of the majority of grains (10–100 $\mu$m) in tungsten of this type. Qualitatively the same picture was also observed for similar distributions determined, while treating the relief data for other mirror specimens.

The calculation procedure and the results obtained were described in work [6] in detail. Here, we only note that, in all cases, the "relief" has some components, the longitudinal dimensions of which strongly exceed the dimension of the largest "crystals".

Hence, the experimental and simulation results testify that even if polycrystalline metallic mirrors with the crystal sizes much less than the reflected radiation wavelength are used, the relief appearing at the sputtering will result in a deterioration of the reflection coefficient. An illustrative example of that can be the emergence of a relief with a typical inhomogeneity size of about 1 $\mu$m on the surface of a mirror fabricated from the crystallized amorphous alloy, although the corresponding crystal size equals 30–70 nm [6].

### *2.2. Single-crystalline mirrors*

The establishment of the reason why polycrystalline mirrors degrade gave us ground to draw a conclusion that it is possible to avoid the formation of a step-like structure on the surface by fabricating mirrors from single crystals. The corresponding experiments were carried out with single-crystalline mirrors fabricated of Mo, W, and stainless steel with various orientations of principal crystallographic axes intersecting the surface: (100), (111), and (110). The specimens were sputtered making use of deuterium plasma ions with a wide energy distribution that was qualitatively similar to the energy spectrum of charge exchange atoms in the ITER. The behavior of single-crystalline mirrors at a long-term sputtering was studied [7, 8], and they were shown to be much more resistant with respect to the relief development at the erosion than the polycrystalline ones (Fig. 6). This result was confirmed experimentally on the active thermonuclear installations Tore Supra (France) [9] and ASDEX (Germany) [10].

On the basis of the presented data, the ITER community adopted single-crystalline molybdenum as a material for the first mirrors for the plasma diagnostics in the ITER. This material has a low enough sputtering coefficient and not very low $R$ in the near ultraviolet and visible spectral ranges.

However, under the ITER conditions, it is highly probable that, under the influence of $\gamma$ and neutron radiation, the single crystal will lose its perfect crystallographic structure (structural defects will emerge). Then the sputtering by CEAs can result in the surface roughness development and the loss of optical properties. This fact is confirmed by the results of experiments on the sputtering of a single-crystalline mirror with a defect structure, when a considerable degradation of $R$ was observed, although it was lower in comparison with the degradation rate of a polycrystalline mirror. At the same time, we are not acquainted with experiments that would combine the influences of $\gamma$ and neutron radiation, as well as ionic bombardment, on the surface of single-crystalline mirrors.

### *2.3. Film mirrors*

We also examined another possibility to avoid the formation of a step-wise structure at the sputtering of a polycrystalline mirror, namely, the coating of the mirror surface with a metallic film possessing a sufficiently high $R$ (Rh, Mo) and a thickness of a few microns that would be sufficient to sustain a long-term sputtering by CEAs at the mirror location site in the ITER chamber. Such films, as a rule, have a fine-crystalline structure ($\leq$100 nm). Therefore, the scale of a microrelief that appears at their long-term sputtering was expected to be much smaller than the light wavelength in the working spectral range of the mirror.

Experiments with various films on various substrates (Be/Cu, Cu/Cu, Rh/Cu, Rh/V, Rh/SS, Mo/SS, and Mo/Mo) testified to a large divergence between their behavior at the exposure to deuterium plasma ions. If the film adhesion to the substrate was strong, the long-term sputtering almost did not induce a deterioration in the optical properties of those mirrors. For instance, Rh/Cu films fabricated by impressing the metal into the substrate kept their characteristics even after a layer more than 7 $\mu$m in thickness was sputtered [7, 8] (see Fig. 6).

However, to obtain films with a high adhesion is an uneasy task. In a lot of cases where the specimens of film mirrors were exposed to a deuterium plasma at room temperature, there appeared blisters at the film–substrate interface [12, 13]. In work [13], the probability of their appearance was reduced by heating up the specimens during their sputtering with deuterium plasma ions to a temperature of about 200 °C. Most likely, such an influence of the heating resulted from an increase in the rate of backward, with respect to the film surface, diffusion of deuterium that penetrates into the film. This result is very important, because it opens an opportunity to use film mirrors in the ITER.

Since the metallic films, as a rule, have a polycrystalline structure, their surface gradually becomes rougher and rougher at the long-term sputtering, so that their optical properties, as a consequence, worsen.

### *2.4. Mirrors fabricated from amorphous alloys*

An alternative to single-crystalline mirrors can be mirrors fabricated from amorphous metallic alloys (AMAs). Unlike the perfect structure of single crystals, AMAs have no structural order already at distances of several nanometers. Therefore, they should preserve the initial quality of their surface during an arbitrary sputtering time. This assumption was made as long ago as 1999 in work [11]. However, it was confirmed only after a technology for producing amorphous ingots with dimensions sufficient for the fabrication of amorphous mirrors ($\geq$10 mm) had been developed.

The results of experiments with mirrors fabricated from amorphous alloys on the basis of zirconium and titanium proved their extreme stability with respect to the long-term sputtering [14, 15]. In particular, a mirror made of the AMA preserved its optical properties even after a layer 13.4 $\mu$m in thickness had been sputtered (Fig. 7). The microscopy data testified that the mirror surface did not changed in comparison with its initial state before the sputtering started.

In the experiments devoted to the study of the interaction between a deuterium plasma and the mirrors fabricated from the amorphous alloy ZrTiNiCuBe, another important result concerning the deuterium absorption at exposing the mirrors to ions with an energy of several tens of electron-volts was obtained [15]. Absorption turned out much more effective than that obtained in the experiments where the saturation with hydrogen was carried out from the gas phase or in an electrolyte. The measured dependence of the absorbed deuterium mass on the ion fluence ($\leq 1.5 \times 10^{25}$ ion/m$^2$) enabled us to estimate

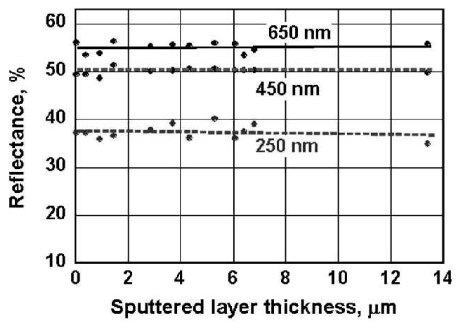


***Fig. 7.*** Dependences of the amorphous mirror reflectance on the thickness of the layer sputtered by argon ions within the energy interval of 0.1–1.35 keV for various wavelengths [14]

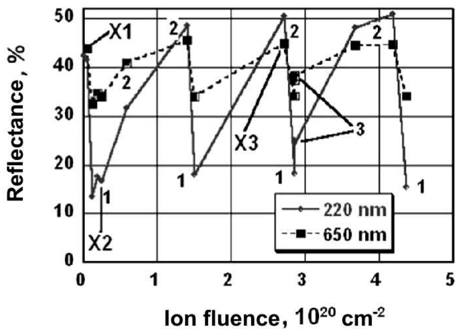


***Fig. 8.*** Reflectance behavior for Be mirror specimens at indicated wavelengths: (*1*) reduction after the bombardment with 1350-eV ions, (*2*) restoration after the long-term exposure to 60-eV ions, (*3*) partial restoration after the annealing in vacuum (2 h, 200 °C) [18]

the deuterium absorption efficiency. It turned out to equal about 10% at an ion energy of about 60 eV. This value is approximately 10 times higher than the corresponding parameter in the experiments on the saturation with hydrogen in an electrolyte [16]. Such a difference results from the capability of hydrogen (deuterium) plasma ions possessing the indicated energies to easily penetrate into the alloy depth to a distance that exceeds the oxide layer thickness (a few nanometers) and at once to find themselves in the material bulk. At the same time, in the case of hydrogen saturation, e.g., from the gas phase, the absorption process is multistage: first, physical absorption of molecules; then, dissociation of molecules and chemical sorption; and, only afterward, diffusion.

A high absorption ability of ZrTiNiCuBe amorphous alloys with respect to hydrogen isotopes gives no chances for them to be used as a material for in-vessel mirrors for the plasma diagnostics in the ITER. However, the rather rapid progress in the development of such materials allows us to hope for that, after a while, there will be created AMA with a negligibly low ability to keep hydrogen and its isotopes, which would allow them to be used as a material for in-vessel mirrors under the condition of high CEA, neutron, and $\gamma$ radiation fluxes.

## 3. Influence of Chemical Processes on Optical Properties of Mirrors

### *3.1. Beryllium mirrors*

It is known that beryllium is chosen as a material for the first wall of the ITER. As the reactor will work, beryllium, owing to its erosion under the influence of a plasma, will propagate over the whole chamber and accumulate at sites the most remote from the plasma confinement volume, including the mirrors installed for optical and laser plasma diagnostics. In other words, there is a high probability that a mirror fabricated from an arbitrary metal, being gradually covered with a beryllium film, will change its optical properties. At the same time, in the case of a Be mirror, the deposition of a Be film should not change its optical characteristics considerably. Therefore, we studied the behavior of Be mirrors under the action of deuterium plasma in detail [17, 18]. The experimental results showed that chemical processes play a considerable role in the modification of optical properties. In particular, this is a transformation of the oxide film into a hydroxide one following the reaction

$$2\text{BeO} + 2\text{D} \Rightarrow \text{Be(OD)}_2 + \text{Be}. \quad (1)$$

Since there will always be a certain amount of oxygen in the ITER, the free Be atom will create an oxide molecule, BeO, again. Therefore, the thickness of the film "oxide + hydroxide" will gradually grow in such a manner. Just this process took place in our experiments with Be mirrors exposed in a deuterium plasma with the oxygen impurity.

Figure 8 demonstrates the repetition of the $R$-reduction process, when the Be mirror is exposed for a short time to keV-range deuterium plasma ions, and the $R$ restoration within a much longer time interval, when the ion energy is low (about 50 eV). For some Be specimens, the procedure "decrease-increase" of the reflectance was repeated 4 or 5 times without any appreciable reduction in the restored level, as one can see from Fig. 8. Such a behavior of $R$ unambiguously testifies that all variations in the optical properties are associated with chemical processes on the surface of a Be mirror, whereas the mirror surface roughness probably remains at the initial level.

The results of x-ray photoelectron spectroscopy (XPS) shown in Fig. 9 allowed us to elucidate the origin of the behavior of Be mirrors, which was observed under the influence of deuterium plasma ions with various energies. Every short-term action of keV-range $D^+$ plasma ions under the conditions of the experimental stand DSM-2 (with a certain amount of water vapor) gives rise to a gradual increase of the oxide layer thickness. The process mainly runs through the formation of hydroxide $Be(OD)_2$ following reaction (1). This first stage of the process is characterized by a complete (or almost complete) transformation of the available oxide film (the extinction coefficient $k \approx 0$) into a hydroxide film (for which $k > 0$ [18]), which results in a reduction of $R$ after a very small fluence of ions in the keV-energy range (at a level of $5 \times 10^{17}$ cm$^{-2}$). The second stage is characterized by a relatively slower growth of the oxide-hydroxide (or maybe only hydroxide) layer thickness owing to the reactions between free Be atoms and new oxygen molecules (with the oxide formation) or water molecules (with the hydroxide formation). This stage is accompanied by a gradual reduction of the $R$ decrease rate and a saturation at ion fluences of about $2 \times 10^{19}$ cm$^{-2}$. The reduction level is deeper for higher energies of deuterium plasma ions. An important factor during the second phase of the growth of the oxide-hydroxide layer thickness is the supply rate of free Be atoms to the layer surface, because neither oxygen nor water molecules can penetrate through the layer. The supply of Be atoms can be provided only at the expense of the beryllium surface sputtering by deuterium ions that passed through the oxide film. The higher the energy of $D^+$ ions, the higher is the sputtering rate of beryllium metal, and the faster is the growth of the oxide-hydroxide film thickness, i.e. the stronger is the reflectance reduction. The saturation of the $R$-reduction is associated with the fact that deuterium ions with a certain energy have a specific path length in the oxygen-containing film.

If we decrease the energy of ions in the course of the mirror exposure to a value, when their path length becomes smaller than the oxide-hydroxide film thickness (in our experiments, the energy of $D^+$ ions was $E_i = 60$ eV), the "source" of free Be atoms turns out switched off (i.e. there is no sputtering of metal volume), and a process opposite to that resulting in the growth of oxide-hydroxide film thickness can begin. This process, which includes a decrease of the oxide layer thickness and the chemical reduction of metallic beryllium, is described by the reaction

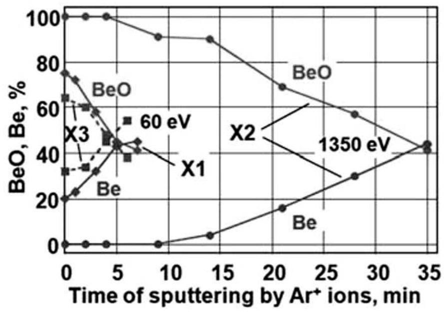


***Fig. 9.*** XPS results for a Be mirror exposed to deuterium plasma ions: (♦) cleaning with $Ar^+$ ions (point X1 in Fig. 8), $E_i = 300$ eV; (•) reflectance reduction after the bombardment with $D^+$ ion, $E_i = 1350$ eV (point X2 in Fig. 8); (■) reflectance restoration after the exposure to $D^+$ ions, $E_i = 60$ eV after the second reflectance reduction (point X3 in Fig. 8) [18]

$$BeO + 2D \Rightarrow D_2O + Be$$

or

$$Be(OD)_2 + 2D \Rightarrow 2D_2O + Be.$$

The rate of metallic beryllium reduction, i.e. the restoration of $R$ to its initial level, is much lower than the rate of $R$ decrease, which is testified by the data shown in Fig. 8.

An important result obtained in experiments with Be mirrors is the fact that two such mirrors underwent several cycles of the reduction and complete or partial restoration of the reflectance $R$ (Fig. 8). The total ion fluence reached a value of $2.5 \times 10^{25}$ ion/m$^2$ for each of them at an average ion current of 15 A/m$^2$ (the total exposure time in deuterium plasma exceeded 60 h). However, the both specimens still preserved a high ability to form the image of a subject, as well as the aluminum standard does [18,19]. This fact is the additional proof that the variations of $R$ are not associated with the development of a microrelief on the Be surface, but completely follow from chemical processes in the oxide film, as was discussed above.

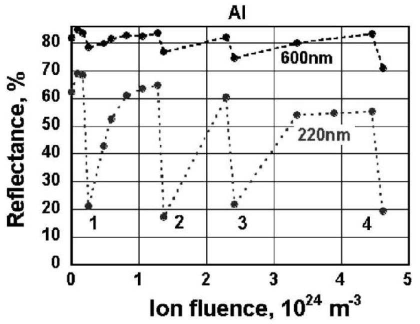


***Fig. 10.*** Dependences of the reflectance at indicated wavelengths on the total ion fluence for an Al mirror specimen. Drastic drops are a result of the short-term bombardment with 1350-eV deuterium plasma ions (stages 1, 2, and 4) and with deuterium plasma ions with a wide energy distribution (100–1350 eV, stage 3). All sections of reflectance growth take place at long-term exposures to low-energy (60 eV) ions

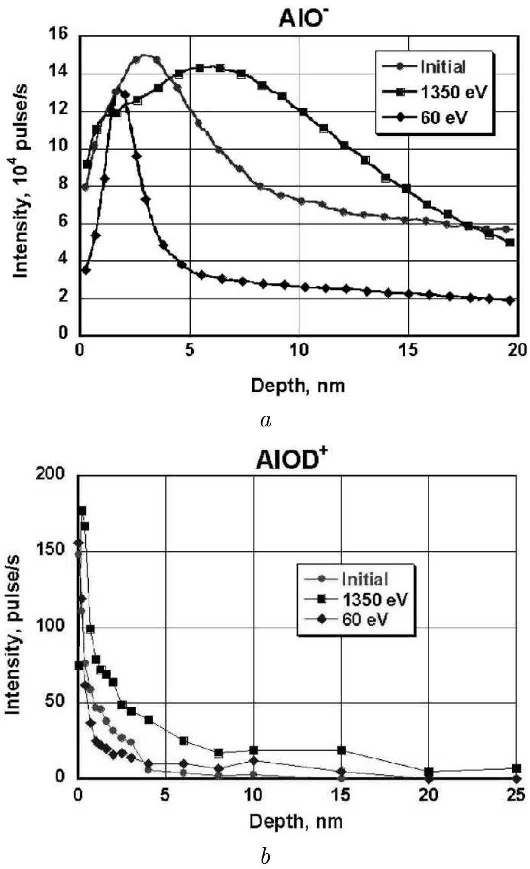


***Fig. 11.*** Results of SIMS analysis for the composition of the near-surface layer in an Al mirror. The data demonstrate the escape rate of $AlO^-$ and $AlOD^+$ ions as functions of their location depth [20]

### *3.2. Aluminum mirrors*

Qualitatively the same effect was observed when exposing Al mirrors under the same conditions, because there exists the so-called diagonal analogy between those elements (Be and Al), which are located close to each other in the Periodic system, i.e. a similarity between a lot of their chemical and physical properties. In our experiments, we demonstrated for the first time that the close analogy is also inherent to the behavior of mirrors fabricated from those metals, when they are subjected to the influence of a hydrogen plasma, as is seen from the comparison of the data presented in Figs. 8 and 10 [18, 20].

The experiments with Al mirrors allowed us to obtain additional proofs in favor of the processes described above for Be mirrors. Using the secondary ion mass spectrometry (SIMS) and Auger spectroscopy methods, the layer-by-layer analysis of the subsurface region in Al mirror specimens after different sequences of operations connected with the influence of deuterium plasma ions on them was carried out [20]. A short-term bombardment with 1350-eV ions was found to increase the thickness of the oxygen-containing layer, whereas a longer exposure to low energy ions led to a considerable reduction of its thickness. In particular, this is evidenced by an increase in the escape of $AlO^-$ and $AlOD^+$ ions, which are fragments of the $Al_2O_3$ and $Al(OD)_3$ molecules, respectively, from the near-surface layer after the specimen has been bombarded with 1350-eV ions and by a considerable reduction in the escape of those fragments after a following, longer exposure to 60-eV ions, which is illustrated in Fig. 11.

Very similar to those distributions over the depth are the data for ions with a mass of 2, which can be both atomic $D^+$ ions and molecular $H_2^+$ ions. However, since deuterium was a working gas, we may assert with a high confidence that the larger fraction of the current created by two-atomic ions is associated with deuterium ions (see Fig. 8 in work [20]).

The measurement results obtained for the escape of $Al_3^+$ clusters from the same aluminum specimens (Fig. 12) are especially illustrative. Such clusters are easily formed when the pure surface of a metal is sputtered. But if the surface is covered with an oxide film, the probability of the cluster formation drastically decreases, whereas the escape of oxygen-containing fragments increases. In our experiments, after a short time of the bombardment with 1350-eV ions, the thickness of the layer, where the gradual growth of the ion cluster escape was observed, considerably increased. However, after the long-term irradiation with 60-eV ions, the transition layer became very thin.

### *3.3. Mirrors fabricated from an amorphous metallic alloy*

Mirror specimens of two types were studied. The both types were fabricated from the same components, but their contents were different: alloy I (Zr(41.2)Ti(13.8)Cu(12.5)Ni(10)Be(22.5), the NSC KhIPT (Ukraine)) and alloy II (Zr(46.75)Ti(8.25)Cu(7.5)Ni(10)Be(27.5), the Hahn Meitner Institute (Germany)) [15]. The both alloys contained Be. Therefore, the behavior of amorphous mirrors fabricated from them was expected to be somewhere similar to that of Be and Al mirrors. The measurements confirmed this assumption. The exposure of mirrors to high- and low-energy ions of a deuterium plasma induced the same variations of $R$ as in the cases with Be and Al mirrors. However, unlike Be mirrors, the dependence of $R$ on the ion energy is much weaker, which is evidently associated with rather low Be content in this alloy.

Changes of the chemical composition in the near-surface layer of mirrors fabricated from the AMA at the interaction of plasma $D^+$ ions with the surface were obtained with the help of the SIMS method. Some of the data obtained are depicted in Fig. 13. Every specimen was analyzed at two sections designated as 1 and 2, and the corresponding results are plotted. The data discrepancies between the sections are rather small, and they can be neglected. The SIMS data testify that, after the bombardment with 1350-eV ions of a deuterium plasma, an appreciable increase in the layer thickness is observed for the oxides of all metals, without exceptions, entering the alloy composition. However, after the irradiation with 60-eV ions, the thickness of the oxide layer almost returns to the value registered immediately after the sputtering with argon ions.

The fact that it is beryllium that plays a key role in the processes of surface oxidation for the mirrors fabricated from amorphous alloys coincides with the results of work [21], where, while analyzing the near-surface layer of specimens with the same composition, the cited authors used the XPS method.

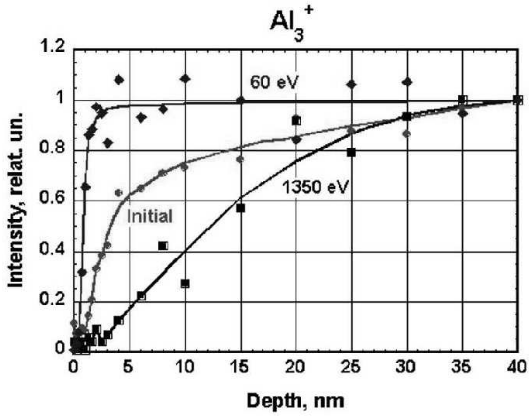


***Fig. 12.*** The same as in Fig. 11, but for $Al_3^+$ clusters

The presented data allow us to understand the results described above that concern the qualitatively similar behavior of the reflectance $R$ in the cases of beryllium mirrors and mirrors fabricated from Be-containing AMAs (Figs. 8, 9, and 13).

## 4. Simulation of Neutron Irradiation

According to the results of calculations carried out many years ago, the defects that are formed in a mirror material by high-energy neutrons can be simulated by bombarding the mirrors with MeV-range ions. To prevent changes in the chemical composition of the mirror subsurface layer, the ions of the same metal, of which the mirror is made, have to be used [8,22]. Therefore, in our experiments, the copper mirror was exposed to a flux of $Cu^+$ ions, the stainless steel mirrors to a flux of $Cr^+$ ions, the aluminum mirror to a flux of $Al^+$ ions with energies of 1–3 MeV, and the tungsten mirrors to a flux of $W^{6+}$ ions accelerated to an energy of 20 MeV. On the basis of the data for the energy distribution of CEAs, both obtained on the PLT, ASDEX, and JFT-IIU tokamaks and found from simulation calculations [1], all the mirrors but the W ones were bombarded with deuterium plasma ions in a wide energy spectrum from 50 to 1350 eV.

In those simulation experiments, it was found that neutron irradiation alone cannot induce appreciable changes in the reflectance $R(\lambda)$ up to doses of about 10 dpa, which considerably exceed doses typical of the ITER within the whole time of its exploitation. Later, this result was qualitatively confirmed in direct experiments with neutron irradiation, when a Mo corner

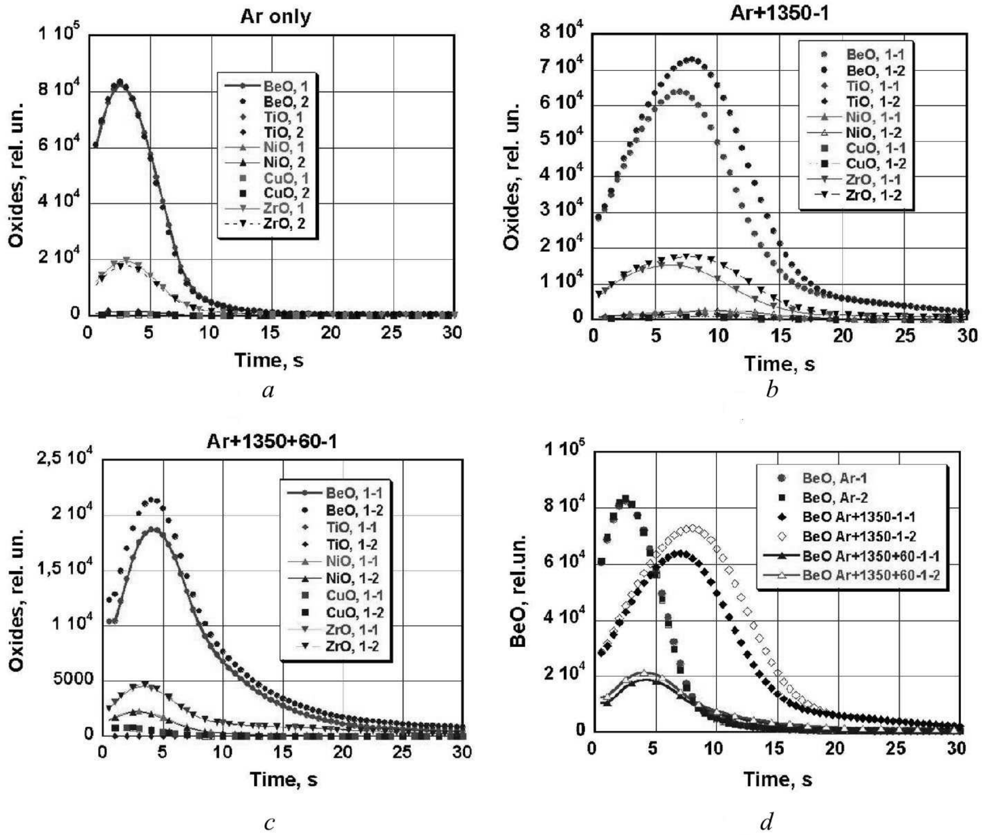


***Fig. 13.*** SIMS data for specimens fabricated from Zr(46.75)Ti(8.25)Cu(7.5)Ni(10)Be(27.5) alloy. Time dependences of the sputtering are shown for all oxides (*a*, *b*, *c*) and only for beryllium oxide (*d*), after the sputtering with $Ar^+$ ions (*a*), after the bombardment with 1350-eV deuterium plasma ions (*b*), and after the exposure to 60-eV ions (*c*)

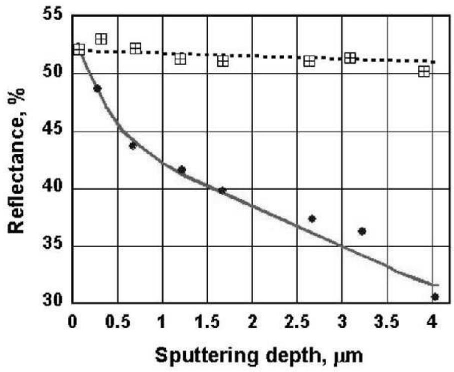


***Fig. 14.*** Dependences of the reflectance at a wavelength of 600 nm on the thickness of the layer sputtered with 600-eV argon ions for tungsten mirror specimens of two types–recrystallized (W-rc) and ITER-grade (W-Ig) ones–non-irradiated (curves) and irradiated with 20-MeV W ions to 3 dpa (symbols)

reflector was exposed for a long time in the Japanese nuclear reactor [23].

However, much more important are researches of the simultaneous influence of neutrons and CEAs. Experiments simulating this situation were carried out with specimens of Cu, SS, and W mirrors. It was shown [5, 8, 11] that, within the interval of neutron irradiation doses that are expected for the ITER (approximately 3 dpa), the degradation of the optical properties of the mirrors at their long-term sputtering with ions of deuterium plasma practically does not depend on whether the mirrors were preliminary bombarded with MeV-range metal ions or not.

As an example, the dependences of the mirror reflectance at a wavelength of 600 nm and the normal light incidence on the thickness of a layer sputtered by 600-eV argon ions are depicted for tungsten mirror specimens of two types (see Fig. 14): fabricated from recrystallized W (W-rc) and ITER-grade W (W-Ig). One side of each specimen was bombarded with 20-MeV W ions, and the other served as a reference non-irradiated specimen. From the figure, one can see that there is no appreciable difference between the behaviors of $R$ for both specimen sides: irradiated with W ions and non-irradiated.

## 5. Bennett's Formula

In order to estimate the surface roughness that appears at the erosion of a metallic mirror, Bennett's formula is often used [24]:

$$R = R_0 \exp\left(-\frac{(4\pi d)^2}{\lambda^2}\right). \quad (2)$$

Here, $R_0$ is the reflectance of an ideally smooth surface at the normal light incidence, $\lambda$ the length of the radiation wave reflected from the mirror surface with the coefficient $R$, and $d$ is the average roughness of this surface. When using this formula, it is often overlooked that it was derived for a surface, the roughness of which can be described by the Gauss formula. Actually, in most cases where the matter concerns the sputtering of metallic mirrors, the surface relief has an absolutely different character. For example, in the case of a polycrystalline material, the surface can be step-like (see Figs. 2, 3, and 5 in this work; Fig. 13 in work [8]; and Figs. 4 and 5 in work [5]); there can appear etching pits (Fig. 2, *b* in work [25]), "crests and valleys" (Fig. 4 in work [6]), and so forth.

The simplest way is to verify whether it is correct to use formula (2) for the roughness estimation. This can be done by applying it formally to various wavelengths. If the found roughness values differ only a little from one another, this will mean that the formula can be used for the description of a relief on the given mirror and that it will provide a rather correct average characteristic of the roughness.

Here are some examples of this approach. In Fig. 15, the typical data obtained for a copper mirror and mirror from stainless steel after their sputtering by 2.5 and 4 $\mu$m, respectively, are shown. The data in Fig. 15 were taken for the same SS mirrors, the degradation of the optical properties of which is illustrated in Fig. 4. For all mirrors, the relief was typically stepwise, with a finer relief observed on separate grains in the copper mirror and the absence of any relief on the grains of the stainless steel mirror (Fig. 13, [8]). The data of Fig. 15 testify that the average roughness of polycrystalline mirrors is not the same at different wavelengths, because the stepwise

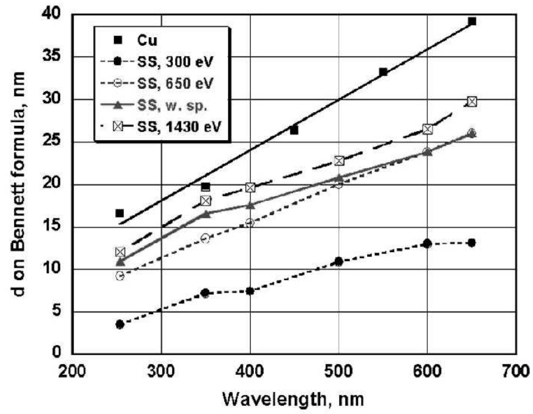


***Fig. 15.*** Average roughnesses $d$ found using formula (2) for copper and stainless steel mirror specimens at various energies of bombarding deuterium plasma ions, and their dependences on the incident light wavelength

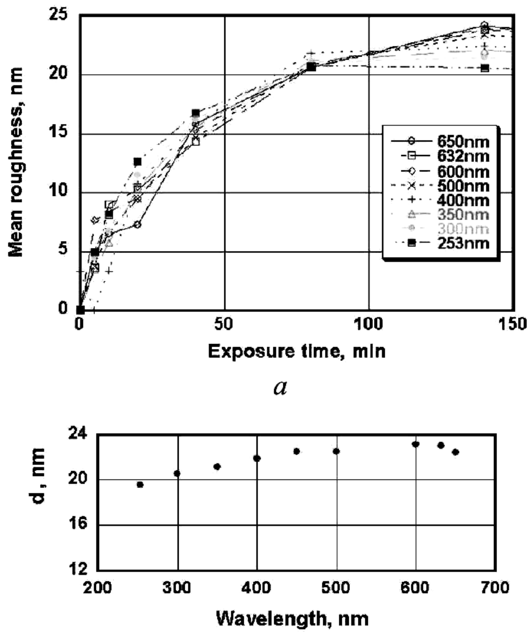


***Fig. 16.*** Average roughness $d$ found using formula (2) for a mirror specimen "the Rh film on the copper substrate" and its dependence on the sputtering time (*a*) and the wavelength after the last exposure in plasma (*b*)

structure of the surface does not satisfies conditions, for which Bennett's formula was derived.

For some examined film mirrors (not for all!), the calculated roughnesses changed within a narrow interval. As an example of a rough surface that can be described by Bennett's formula, Fig. 16 demonstrates the values of the average roughness calculated at various wavelengths for one of the specimens "the Rh film on the copper substrate". One can see that, for this specimen, the roughness of the indicated type started to form already at the early stage of the sputtering (Fig. 16, *b*). Other examples when the roughness values obtained for similar film specimens at various wavelengths are in satisfactory agreement are described in work [26]. It is evident that such a roughness character cannot be predicted beforehand, without carrying out sputtering.

## 6. Conclusions

For many years of the researches dealing with the behavior of metallic mirrors under conditions similar to those in the thermonuclear reactor ITER, a large body of data was accumulated. The majority of them were presented above. At the beginning of the researches, the bases of the methodology aimed at the optimal choice of a material for in-vessel mirrors used for optical and laser plasma diagnostics in the experimental thermonuclear reactor ITER were created, and the major factors responsible for a deterioration of the optical properties of mirrors were carefully analyzed. On the basis of the elaborated methodology, some experiments that simulated the behavior of mirrors for the plasma diagnostics in the ITER were carried out. The essence of the methodology consists in that, instead of the bombardment with charge exchange atoms in the reactor, the bombardment with deuterium plasma ions of a wide energy distribution qualitatively similar to the spectrum of CEAs is used. Instead of the neutron irradiation, the bombardment with MeV-range ions of the same metal that the mirror is fabricated from is applied.

Systematic simulation researches of the behavior of mirror specimens fabricated from various metals and possessing different structures – polycrystalline mirrors (Be, Al, SS, Cu, Ti, Mo, W, and Ta), single-crystalline mirrors (SS, Ni, Mo, and W), film/substrate mirrors (Be/Cu, Cu/Cu, Rh/Cu, Rh/V, Rh/SS, and Mo/SS), and mirrors fabricated from amorphous alloys of the ZrTiCuNiBe type – under their long-term sputtering with hydrogen and deuterium plasma ions were done. A considerable advantage of the mirrors fabricated from amorphous alloys with respect to their resistance to the roughness development at the long-term sputtering in comparison with the mirrors of other structure types was revealed. This phenomenon stems from the absence of any ordered structure with a scale more than a few nanometers on the surface of amorphous mirrors.

Based on the analysis of the results of numerous experiments with the mirrors fabricated from polycrystalline metals characterized by different structures, a model describing the roughness evolution at the long-term sputtering was developed. Experiments simulating the influence of the simultaneous irradiation with neutrons and charge exchange ions on the metallic (Cu, SS, and W) mirrors, which are promising to be used as in-vessel mirrors for the plasma diagnostics in the ITER, were done.

While studying the interaction between a hydrogen plasma and the Be, Al, Mo, and W mirrors, the mechanisms of chemical processes on the metal surface were established. Those processes give rise to the changes in the optical properties of mirrors, irrespective of whether the ions of a pure hydrogen plasma or the ions of plasma with oxygen impurities are engaged.

The most important results of reported researches were confirmed in the mutual experiments on such large thermonuclear installations as the TEXTOR (Jülich, Germany), Tore Supra (Cadarache, France), and TRIAM-1M (Kyoto, Japan) tokamaks, the heliotron Large Helical Device (Toki, Japan), and a special stand at the Institute of Physics, University of Basel (Switzerland). A conclusion concerning the absence of the influence of neutron irradiation on the optical properties of metallic mirrors, which was drawn on the basis of our simulation experiments, was confirmed in experiments with a Mo corner reflector on the Japanese experimental nuclear reactor. The efficiency of the method proposed for the cleaning of metallic mirrors from contaminating carbon layers with the use of a low-temperature hydrogen plasma and multiply applied in simulation experiments was later confirmed on the toroidal installation TOMAS in Jülich (Germany).

A modification of the optical properties of Be and Al mirrors as a result of the chemical processes invoking under the action of a plasma containing hydrogen and oxygen ions directly concerns the behavior of Be mirrors on the board of satellites that rotate around the Earth: they permanently undergo the action of hydrogen atoms, as well as hydrogen and oxygen ions, available in the upper atmosphere.

The results obtained testify that some of the examined materials possess certain advantages, if being used as diagnostic mirrors under specific environmental conditions in the vacuum chamber of the ITER.

*The authors express their large gratitude to M.G. Nakhodkin, who was an initiator of a series of researches devoted to the dependence of the optical properties of diagnostic mirrors for plasma diagnostics in the ITER on the structure of their prototype materials.*

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ПОВЕДІНКА МЕТАЛЕВИХ ДІАГНОСТИЧНИХ ДЗЕРКАЛ ІЗ РІЗНОЮ СТРУКТУРОЮ В УМОВАХ, ЩО ІМІТУЮТЬ УМОВИ ЇХ РОБОТИ В ТЕРМОЯДЕРНОМУ РЕАКТОРІ ІТЕР

Р е з ю м е

У статті V.S. Voitsenya *et al.* (Plasma Phys. Rep. **20**, 217 (1994)) створено основи методології щодо пошуків оптимального вибору матеріалу внутрішніх дзеркал для оптичних і лазерних методів діагностики плазми в експериментальному термоядерному реакторі ІТЕР. На основі розробленої методології проведені системні імітаційні дослідження при довготривалому розпиленні іонами дейтерієвої (у деяких випадках аргону) плазми поведінки зразків дзеркал із різних металів та із різною структурою: полікристалічною (Be, Al, SS, Cu, Ti, Mo, W, Ta), монокристалічною (SS, Ni, Mo, W), плівкових (плівка/підкладка, тобто Be/Cu, Cu/Cu, Rh/Cu, Rh/V, Rh/SS, Mo/SS, Mo/Mo) та дзеркал із аморфних сплавів типу ZrTiCuNiBe. Показано значну перевагу дзеркал із аморфних сплавів у стійкості до розвитку шорсткості при довготривалому розпиленні в порівнянні з дзеркалами іншого типу структури, що зумовлено відсутністю будь-якої упорядкованої структури на рівні більш ніж декілька нанометрів. Найбільш важливі результати цих досліджень знайшли підтвердження в експериментах, спільно проведених у зарубіжних центрах на крупних термоядерних установках: токамаки TEXTOR (Юліх) і ASDEX-U (Гарчінг) в Німеччині, Tore Supra (Кадарах, Франція), TRIAM-1M (Кіото, Японія), геліотрон Large Helical Device (Токі, Японія) та установки в університеті Лозанни (Швейцарія) та в місті Юліх (Німеччина).