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ORIGINAL PAPER

Modeling of the behavior of in-vessel mirrors for ITER with ECR plasma discharges

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Abstract. The main experimental results related to the problems associated with in-vessel mirrors in ITER obtained with the DSM-2 facility at the Kharkov Institute of Plasma Physics over the past few years are described and discussed. Mirrors made from various polycrystalline (Be, Al, SS, Cu, Mo, Ta, W) and single crystal (Ni, SS, Mo, W) metals, metal films (Be, Cu, Mo, Rh) on different metal substrates (V, SS, Cu, Mo), and an amorphous alloy (ZrTiCuNiBe) have been studied. In addition, the behavior of protective oxide coatings under plasma bombardment has also been analyzed.

Key words: ECR plasma • mirrors for plasma diagnostics in ITER • structure • sputtering • chemical erosion

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Introduction

Super-high frequency (SHF) plasma discharges, based on the electron cyclotron resonance (ECR) are widely used in technical applications of plasmas. This type of discharge is steady state, electrodeless, and provides a wide variation of plasma parameters which can satisfy different experimental requirements. An additional advantage of ECR discharges in comparison to many other types of plasma discharges (e.g., reflex discharges), is that they can be spatially separated from the specimen. Thus plasma parameters are nearly independent of procedures such as biasing of specimens.

For many years, such discharges have been used at the Kharkov Institute of Plasma Physics (KIPP – Kharkov, Ukraine) to simulate the effects of a fusion reactor environment on the in-vessel mirrors which are a necessary part of many plasma diagnostic systems. As such mirrors would not be directly exposed to fusion plasmas, the most serious effect will likely be due to bombardment by charge exchange atoms (CXAs) and due to deposition of plasma impurities. The most likely deposits would originate from plasma-facing components of the reactor; most importantly, the first wall, which will be made of Be in ITER (International Thermonuclear Experimental Reactor). In this paper we present an overview of the facility, and a summary of the simulation experiments performed over the past decade.

Details of the experiment

Plasma discharges are produced with a double-mirror magnetic configuration as shown in Fig. 1 of [4] and

Fig. 7 of [14]. The stainless steel vacuum chamber of 1 m long and 0.5 m in diameter has two axial ports (0.23 m in diameter) where the magnetic coils are located. The magnetic field strength is 0.5 kG at the chamber center and 2.25 kG in the mirror regions. The chamber is excited as a multi-mode cavity resonator by means of an antenna in the form of a rectangular waveguide, situated perpendicularly to the axis. The antenna is connected to the SHF generator through a teflon window and copper waveguide. The input power can be varied continuously from 0 to 1000 W. Typical plasma parameters with 200–400 W input power at 2.375 GHz, and with a continuous flow of the deuterium working gas with pressure 2×10^{-5}–10^{-3} torr were: $n_e \sim 10^{10}$ cm^{-3}, $T_e \sim 5$ eV.

Test specimens were mounted on a water-cooled movable holder which could be introduced into the vacuum chamber through an air-lock. The holder was placed on the system axis, just outside the magnetic mirror. A negative bias voltage from 0.05 to 1.5 kV may be applied to the specimen holder to accelerate plasma ions to the specimen surface. The bias voltage can either be kept constant, or made to oscillate within this range with a frequency of 100 Hz. The mean ion current density to specimens was typically ~ 10 A/m^2. The existence of D^+, D_2^+ and D_3^+ [8] in the extracted ion species means that there is a heavier weighting of low-energy D atoms/ions in the distributed energy experiments. Thus, the nature of the energy distribution with the oscillating bias qualitatively corresponds to the energy spectrum of CXAs measured in the tokamaks TEXTOR [17] and JFT-2U [12], and calculated for ITER [6]. This is an important factor in comparing the present experiments with those employing a fixed ion energy.

Specular reflectance in the wavelength range λ = 220–650 nm was measured at normal incidence by means of the two channel methods described in [14]. Prior to any reflectance measurements, specimens were first biased to 50–60 V, to provide a "soft" plasma-chemical cleaning. This cleaning procedure removed residual contaminants from washing and storage. A similar cleaning procedure with low-energy ions has also been used to remove contaminating films from mirrors exposed in fusion devices or deposition experiments; see section "Removing deposited films from mirrors". For some specimens, ellipsometry was used to determine the optical characteristics of mirrors following plasma exposure.

The primary type of experiment performed was to measure the changes to the spectral reflectivity, $R(\lambda)$, as a function of the thickness, h, of the eroded layer. The thickness of the sputtered layer was determined by measuring the mass loss (Δm) of the test specimen, with an accuracy of ~ 25 μg. *Ex-situ* measurements of $R(\lambda)$ and Δm were made after each ion-exposure step to determine the $R(h)$ dependence. In several cases, the measurements were specifically checked to show that the thickness of the sputtered layer was approximately proportional to the total ion fluence.

In addition to spectral reflectivity and mass-loss measurements, scanning electron microscopy (SEM) images could be taken to show the development of surface micro-relief with increasing thickness of the sputtered layer. The micro-relief leads to a decrease in the specular reflectance and an increase in diffuse reflectance. Thus, the degradation of optical properties results in both the distortion of the transmitted image, and a decrease in the reflected light intensity.

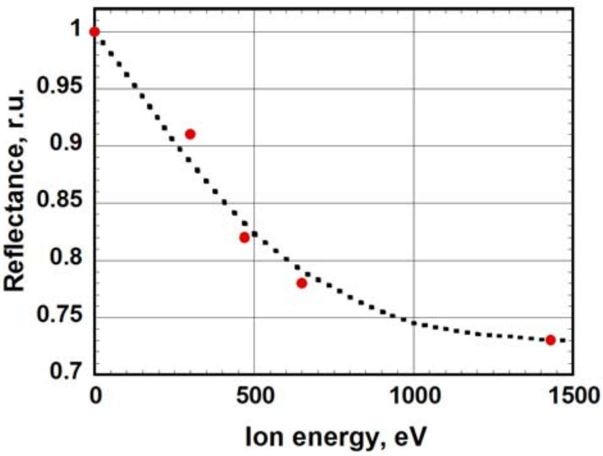

Fig. 1. The degradation of reflectance of SS mirrors after a 4 μm surface layer was removed by sputtering by ions from a deuterium plasma. The point at E_i = 470 eV corresponds to experiments with a wide energy distribution having, according to calculations, a mean ion energy of ~ 470 eV.

Results

Polycrystalline mirrors

Polycrystalline (pc) mirrors of Be, Al, SS, Cu, Mo, Ta, and W were investigated. One of the most interesting features of these studies was the observation of a step-like structure developing on the surface [1, 3, 4, 14], due to the different sputtering yields on grains with different crystallographic orientations. It was found that for Cu and SS mirrors, the development of surface roughness, for a given eroded depth, increased strongly with increasing ion energy. Figure 1 shows the reduction in reflectance found for three identical SS mirrors which were each sputtered to the same depth (4 μm) using 300, 650 or 1430 eV ions. A fourth specimen was eroded using a wide energy distribution having a mean ion energy of ~ 470 eV. In this latter case, the sputtering rate corresponded approximately to the mean ion energy.

The important conclusion from these experiments is that the difference in sputtering yields for different crystal orientations of grains increases with increasing ion energy.

The results obtained with pc mirror specimens led directly to the idea of using single-crystal metal mirrors in locations where sputtering by CXAs dominates over deposition [21].

Single crystal (sc) mirrors

Figure 2 compares the reflectance of pc and single crystal (sc) mirrors with a (111) orientation as a function of eroded layer thickness. The ions had a wide energy distribution. Even when the eroded layer exceeds 5–6 μm, there is very little degradation of the reflectance

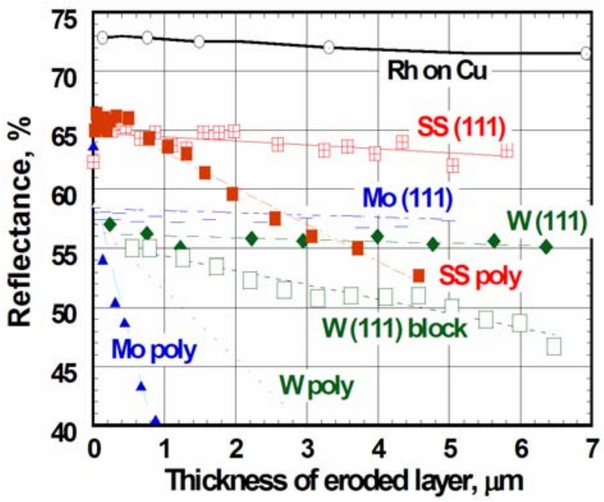

Fig. 2. Reflectance at λ = 600 nm as functions of the thickness of the eroded layer under ion sputtering for sc and pc of SS, Mo and W mirrors. W (111) block denotes the data for a pc tungsten consisting of (111) subdomains with an orientation within approximately 2° relatively to the main direction.

for sc mirrors. Similar behavior was observed for sc mirrors with other ((100) and (110) [14]) orientations. For the sc W mirror, the very low sputtering yield means that the total ion fluence of 7×10^{25} ions/m^2 was required to achieve the final eroded depth, 6.4 μm. Such a large fluence corresponds to the CXA fluence of $\sim 5 \times 10^3$ ITER pulses (CXA flux: 3×10^{19} at/m^2·s, pulse duration: 400 s). The advantage of sc over pc mirrors has been qualitatively confirmed in experiments in the TEXTOR tokamak [10].

Metal film mirrors

Metal film mirrors with a micron-scale thickness are polycrystalline materials, but the size of their crystallites is much smaller than the wavelength of visible light. Thus, in principle, even long-term sputtering should not result in the development of the type of micro-relief observed for bulk pc metals. Such mirrors could, therefore, be more resistant to a degradation in reflectance. Experiments have generally confirmed this idea. As an example, reflectance data for a Rh film on a Cu substrate is also shown in Fig. 2. Mirrors with other metal films (Be, Cu, Mo, Rh) on various metal substrates (V, SS, Cu, Mo) showed similar results. In the case of a Cu film on a Cu substrate, the lack of degradation of reflectance was observed for the complete removal of the film.

Be mirrors

The principal reason for considering Be mirrors is the fact that Be has been chosen as the material for the first wall of ITER (eg., [7]). Be atoms eroded due to plasma contact will inevitably be redeposited on various other surfaces, including those remote from the plasma, such as first mirrors. According to rough estimates, a Be film thickness of ~ 20 nm would transform the reflectance of a mirror of any metal to values typical of a bulk Be mirror. Thus an understanding of the behavior of Be mirrors in an ITER-like environment will be important regardless of the material chosen for the initial mirror surface. As Be is more chemically active than many other metals, a consideration of residual background gases of carbon- or oxygen-containing molecules in addition to hydrogenic CXAs is also required.

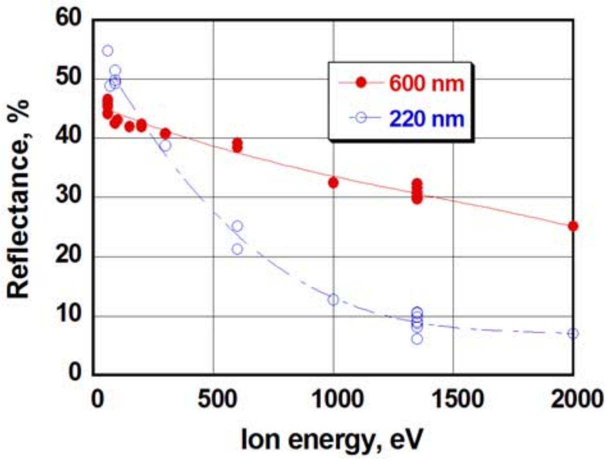

Fig. 3. Degradation of Be mirror reflectance as a function of the energy of deuterium plasma ions.

In the experiments with Be mirrors, a sharp decrease in reflectance in the wavelength range 220–650 nm was observed every time such mirrors were subjected to high-energy ions from deuterium plasmas [2].

This decrease in reflectance (Fig. 3) increases strongly with ion energy, particularly at shorter wavelengths, and is not accompanied by any measurable mass loss. Surprisingly, the reflectance can be fully restored by subsequent exposure of the mirror to a much larger fluence of low-energy (50–60 eV) ions from the same plasma (see Fig. 4, stage 2–3). Further investigation has shown that this behavior is due to a transformation of the normal oxide layer on the Be surface into a hydroxide film. Ellipsometry data show a significant increase of both the refraction and extinction indices of the transformed film as compared with BeO [2].

Amorphous metallic alloy mirrors

Because of their high structural homogeneity, amorphous metallic alloy (AMA) mirrors have the potential to be similar to sc mirrors in their resistance to the development of surface roughness due to sputtering. Mirrors fabricated from two ZrTiCuNiBe alloys were investigated by exposure to ions from deuterium and argon plasmas [5, 19]. In the argon plasma experiments, two mirrors were fabricated from one molded billet; however, one of the mirrors was vacuum annealed at 773 K for 1 h to transform the amorphous structure into a fine crystal structure (prior to polishing). Under long-term ion exposure, the surface of the amorphous mirror remained smooth (Fig. 5a), with no change in reflectance to a sputtered depth of > 13 μm (Fig. 6). On the other hand, the recrystallized mirror became very rough (Fig. 5b) [19], and experience significant loss of reflection.

The inclusion of Be in the alloys had the effect of creating a response to deuterium plasma ions similar to

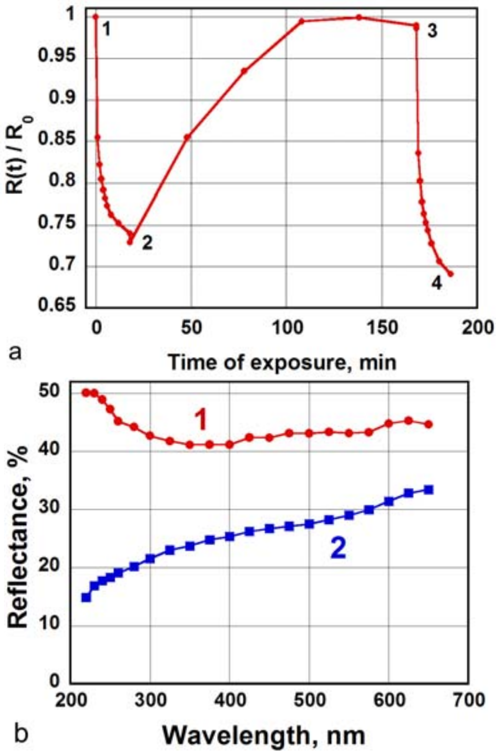

Fig. 4. (a) Relative value of reflectance (at λ = 650 nm) measured *in-situ* [2] as a function of the exposure time to ions from a deuterium plasma; parts 1–2 and 3–4 which show a reflectance drop are a result of exposure to ions with energy 1.35 keV, the part 2–3 is the restoration of reflectance due to exposure to ions with energy 50 eV. The ion current density is 16.5 A/m^2. (b) Spectral reflectance of the Be specimen before (1) and after the first drop in reflectance (2).

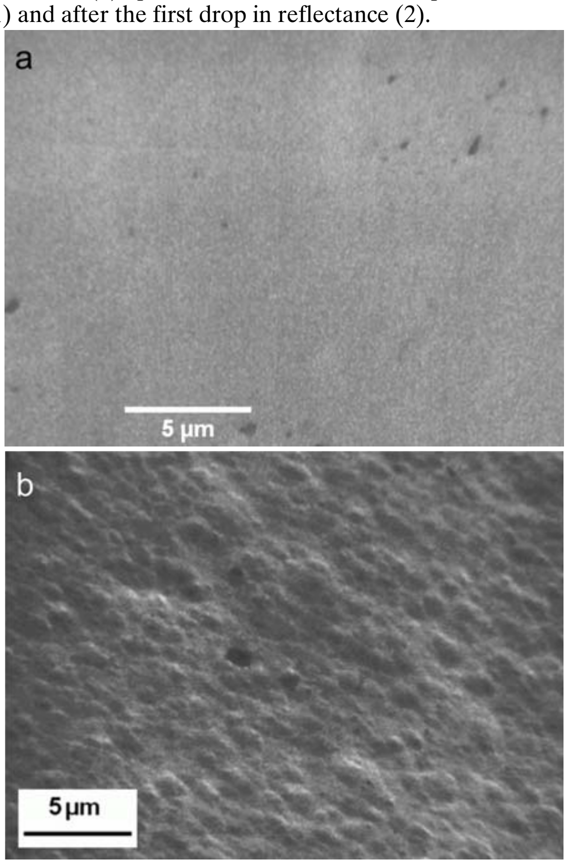

Fig. 5. SEM photos of amorphous (a) and crystallized (b) mirrors made from the same mold after sputtering with Ar$^+$ ions (energy 150–1350 eV) [19].

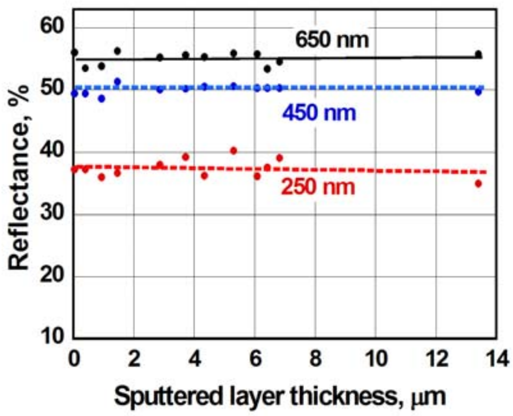

Fig. 6. Reflectance of amorphous mirrors vs. thickness of the sputtered layer at three wavelengths [19].

that observed for pure Be mirrors; i.e., the reflectance decreased following bombardment with keV-energy ions (due to the development of a thick oxide/hydroxide layer), and recovered following exposure to 50–60 eV ions (which remove the layer) [18]. The only difference is that the decreases in reflectance following exposure to keV-energy ions were much smaller in comparison to the pure Be mirrors, probably due to the small fraction of Be (22.5%) in the alloy. An example of the reflectance behavior of an AMA mirror is shown in Fig. 7. The data labeled 1350 eV were measured following a brief (fluence 6×10^{23} m^{-2}) exposure to the energetic ions, while the measurements labeled 60 eV were made following a longer 60 eV ion exposure (fluence 2.1×10^{24} m^{-2}) which was subsequent to an energetic ion exposure.

Removing deposited films from mirrors

The same plasma facility (DSM-2) has been used to perform experiments on cleaning mirrors of carbon-containing films. Films, ranging in thickness from ~ 10 nm to ~ 1 μm were deposited on SS or Mo mirror substrates in other laboratory facilities, or in fusion devices (LHD, Tore Supra, TEXTOR, TRIAM-1M). It was found that pure C-H films could be effectively

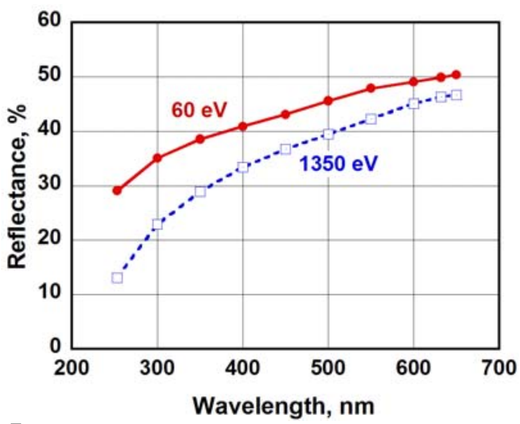

Fig. 7. The reflectance of an amorphous mirror specimen after exposure to 60 eV (solid circles) and 1.35 keV (open squares) ions from a deuterium plasma. For this particular specimen, the drop of reflectance due to the energetic ions was repeated three times and restoration after long-term exposure to low energy ions from the same deuterium plasma was repeated twice.

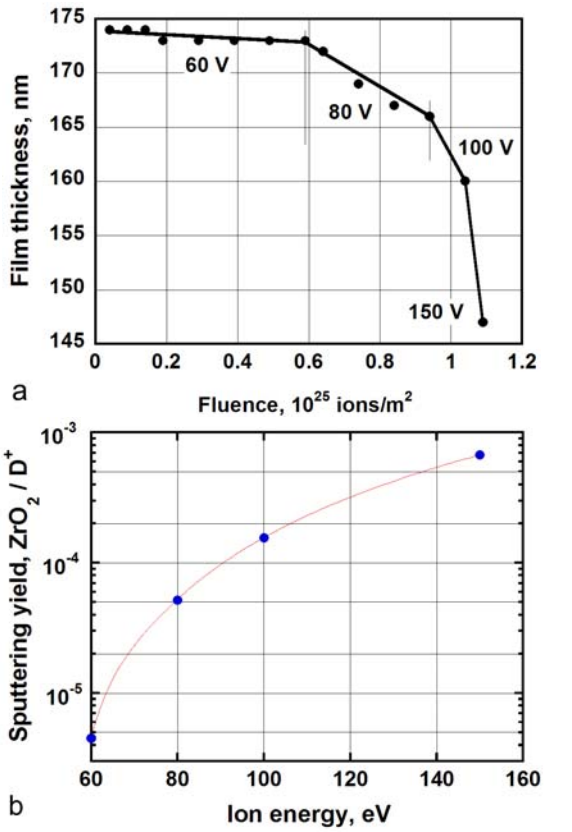

Fig. 8. Dependence of ZrO_2 film thickness on the exposure time to deuterium plasma ions of the indicated energies (a), and a sputtering yield as the function of ion energy (b) found from (a).

cleaned with deuterium plasma ions with energies as low as ~ 15 eV [20]. However, to remove mixed carbon--metal films (LHD, Fig. 6 in [22]; TRIAM-1M, Fig. 7 in [11]), the ion energy had to be increased to 60 eV or greater. The rate of film removal for the thick carbon films (≤ 1 μm) was estimated by measuring the shift of lines in an interference pattern [9].

Erosion of oxides

A recent suggestion [13] for the divertor diagnostics in ITER is to use mirrors of highly reflective metals (Al, Ag) deposited on a Si substrate protected by a dielectric film (Al_2O_3, ZrO_2). The reflectance of such mirrors in the visible and near-IR was found to be only weakly sensitive to the deposition of hydrocarbon films, up to a thickness of ~ 100 nm. However, the deposition of pure carbon films, which have a much higher extinction coefficient, can lead to a significant decrease in reflectance. In the ITER divertor with CFC target plates, such a situation is quite probable and thus special experiments were performed to test the prospect of using a local discharge for *in-situ* cleaning of such mirrors. Part of this process involved testing of the resistance of such dielectric films to erosion by plasma ions.

As shown in Fig. 8 [15], ZrO_2 coatings experienced very little erosion due to ~ 60 eV deuterium ions, while such ions can be very effective at removing carbon films. In addition, it was shown that even after sputtering ~ 100 nm of the amorphous ZrO_2 layer, the oxide layer remained smooth, and no increase in diffuse reflection was observed.

As part of the experiments which involved exposing Be, Al, and W mirrors as well as the ZrO_2 protected Al film mirror specimens to the ECR plasma ions, it has been possible to estimate the efficiency with which the oxygen from the surface oxide layer has been removed. These data are presented in Table 1 together with the data from a paper by Yu. Sakamoto and Yu. Ishibe [16]. It was found that the ZrO_2 film is much more resistant to removal by ion impact than many other oxide layers.

Conclusion

The DSM-2 facility at IPP NSC KIPT, which produces an ECR plasma discharge in a double magnetic mirror configuration, is a very useful tool in the investigation of the behavior of in-vessel plasma diagnostic mirrors. The degradation of optical properties was found to have a strong dependence on many factors, including the structure of the material (pc, sc, film, AMAs), ion energy, chemical composition of the plasma, chemical properties of the mirror material, etc. In addition, low--energy hydrogen plasma ions have been shown to be effective at removing carbon-containing deposits from mirror surfaces, leading to the possibility of using *in-situ* plasma cleaning to restore the reflectivity of mirrors in locations of net deposition.

Table 1. Efficiency of oxide removal by H plasma produced by ECR discharge from [16] and estimated using the data obtained in experiments in the DSM-2 stand with deuterium plasma [2]

Metal	Oxide	*d* (nm)	Time (h)	Efficiency, Oat/10^3 Hat	Reference
Cu	Cu_2O	150	0.02	~ 750	[16]
Ni	NiO	209	3	7.9	[16]
Fe	Fe_2O_3	153	5	3.3	[16]
Mo	M_xO_y	~ 100*	6	~ 3*	[16]
Ti	TiO_2	177	15	2	[16]
Be	BeO	~ 17	1.5	~ 1	[2]
Al	Al_2O_3	~ 8		~ 0.5	****
W	WO_3	~ 10	2	~ 0.5	****
Zr	ZrO_2	~ 1	18	~ 0.01**	****
Zr	ZrO_2	~ 6	3.3	~ 0.3***	****

* Estimated from the data presented in the paper [16].
** Ion energy 60 eV.
*** Ion energy 100 eV.
**** Recent data.

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