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Effect of exposure inside the LHD vessel on optical properties of stainless steel mirrors

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Abstract.
Three stainless steel mirrors were exposed inside the LHD vacuum vessel during the 3-rd experimental campaign. Then the change of spectral reflectance, R(λ), at normal incidence for λ=200÷700 nm was measured. Mirrors in the divertor region and in the diagnostic port became coated with carbon-based films but mirror closest to the plasma confinement volume became cleaner than was initially. The characteristics of films were obtained by different techniques that are in practice for analyzing the near-surface layers. After that the films were slowly removed by series exposures of mirrors to low energy ions of deuterium plasma with measurements of R(λ) after every exposure. In the present paper the comparison is made on reflectance behavior during cleaning procedure with results of reflectance calculations in the framework of the model: the half-transparent film on the stainless steel substrate. The n(λ) and k(λ) data for carbon-based films were taken from previous measurements and from literature.

Keywords: mirror, reflectance, sputtering, surface analysis, contamination, LHD.

1. Introduction

The working efficiency of in-vessel mirrors of diagnostic systems for measuring plasma parameters in a fusion reactor will depend both on the mirror material and on the mirror location inside the reactor vacuum vessel. Till recently the data necessary to predict the behavior under a fusion reactor environment of mirrors fabricated of different metals with different structure (i.e., polycrystal, single crystal, metal film on metal substrate) were obtained in simulation experiments only [1]. The first attempt to study the relationship between the mirror location and modification of mirror optical properties was made for stainless steel mirror samples exposed inside the Large Helical Device (LHD) [2] during one experimental campaign. In this paper the main results obtained after investigation of mirror samples taken out of the LHD vessel are presented.

2. Experimental setup and initial data

Three mirrors of stainless steel type similar to 316 steel, with the size of 20x10x1 mm were mechanically polished and rinsed in an ultrasonic bath filled with acetone. The spectral reflectance at normal incidence of samples was measured in the wavelength range 200-700 nm, and afterward they were installed inside the LHD vacuum vessel in positions shown in Fig.1, where locations of mirror samples are indicated by numbers 1, 3, and 5. The mirror samples were not protected from contact with the plasma produced by a glow discharge during regularly repeated conditioning procedures. Close to these mirror samples the carbon and SS collectors were also disposed, and results of detail surface analysis of these collectors are presented in [2] and [3]. The samples were exposed during the whole 3rd campaign with main peculiarities of operating regimes described in [3, 4]. We mark here only

the most important common characteristics of experiments during that campaign: (i) all entire regions of divertor striking points were covered by graphite tiles in such a way that the plasma of divertor flows was interacting with graphite targets only, (ii) about 80% of discharges were initiated when magnetic axis of the magnetic configuration was positioned at R=3.6 m, (iii) the working gas was H_2 or He with approximate ratio of numbers of working discharges (shots) $N_H/N_{He}\approx0.62$ of the total $N_H+N_{He}\approx10000$, (iv) the total time of glow discharge cleaning (two anodes), about 2300 hrs, was equally distributed between discharges with He and H_2 backgrounds. Plasma was heated by the ECH (~0.55 MW), ICRF (1.5 MW), and NBI (~4.5 MW) methods and the maximal stored energy reached ~0.88 GJ.

It is seen from Fig.1 that the samples were fixed at very different locations: #1 was positioned near the divertor region, #3 – close the plasma border, and #5 – quite deeply in the diagnostic port in the same poloidal cross section and in the same plane as #1 (central plane). After samples have been removed from the vacuum vessel the reflectance was again measured in the same way, and the surface of samples was analyzed by several methods: Auger electron spectroscopy (AES), ion backscattering technique (RBS) using 1.5 MeV He^+ ion beam, scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), profilometry, and ellipsometry at the wavelength 632.8 nm.

It was found that after exposure in LHD the reflectance of all samples has changed from the identical initial level, as is seen from data in Fig.2. The reflectance of mirrors #1 and #5 dropped strongly whereas the reflectance of sample #3 increased significantly. The decrease of samples #1 and #5 reflectance is due to appearance of the contaminating films, which could be easily seen in the white-dark photo of samples, Fig.3. From the character of spectral reflectance (Fig.2) one can conclude that the contaminating film on the #5 sample is thicker than the film on the #1 sample.

The rise of reflectance of the sample #3 after exposure in the LHD vessel we attribute to a not entire cleaning of all three mirrors before they were installed inside the LHD vessel from contamination by some organic film having been deposited due to rinsing samples in an ultrasonic bath (in acetone). It was found by special experiment that the best cleaning of such an organic film can be reached when the rinsing procedure is followed by exposing to impact of low energy hydrogen (deuterium) ions of a ECH discharge plasma, what was not done in the case of these particular mirror samples, #1, #3, and #5. Thus, the increase of reflectance of the sample #3 can be explained by full cleaning of the mirror surface from residuary organic film due to plasma impact, for example, through bombardment by charge exchange atoms during working discharges and by ions during glow discharge conditioning.

The high level of reflectance for mirror sample #3 after removing from LHD vessel was supported by quite high surface quality as was observed by analyzing the SEM photos (not shown in this paper). The data of profilometry measurements (Fig.4) demonstrate that bombardment of the #3 mirror not only cleaned its surface from organic film but also resulted in sputtering erosion of the mirror body. This sample, as others, was fixed to holders by narrow angle bars in such a way that the narrow zones, of ~0.5 mm wide, along both sides of samples were protected from bombardment by projectiles. In Fig. 4 one of protected zones is shown as the area going along the sample between x=0 and x=500 mm (X – is the horizontal axis), and one can see the existence of a small step along the border separating the parts which was protected by the angle bar (left side of the picture) and the other, unprotected

part of sample. This step is also seen on the structure of microrelief (lower part of Fig. 4) measured along the line shown on the area subjected to profilometry. The estimation "on eye" gives that layer with approximately 0.15 μm in depth was eroded on average.

The composition of the contaminating films that appeared on samples #1 and #5 was estimated using AES and RBS data. The RBS data for all three mirror samples together with data for several other samples are presented in Fig.5. According to AES analysis, the deposited layer on the surface of #1 sample was found to consist mainly of C (~40 atomic %) and Fe (~40 atomic %) but on the #5 sample – the only contaminant registered was carbon (~90 atomic %) [2, 3]. The surface of the sample #3 was practically free of carbon however a quite small trace of heavier metal, possibly, copper was registered by RBS. The thickness of deposited layer, according to RBS estimation, was ~100 nm for #5 sample and ~40 nm for the sample #1.

The optical characteristics of film deposited on sample #1 was obtained by ellipsometry at the wavelength 632.8 nm. The calculations of optical indices (n_f, k_f) and the film thickness were done within a simple approximation: a homogeneous film on the SS substrate. As the n and k values of the SS substrate, the indices measured for the sample #3 were used. The refraction and extinction indices of the deposit evaluated with such an approximation are n=2.5 and k=0.33, and the film thickness was estimated as ~26 nm, what is in agreement with RBS data. For sample #5 similar data could not be obtained for the full deposit thickness because of very low reflectance at the wavelength of measurement (see Fig.2).

To know more about properties of contaminating films on samples #1 and #5 and of the quality of mirror surface under the coatings, the cleaning of these films using low energy ions of deuterium plasma was provided. The results of this experiment are presented in the next section.

3. Cleaning the mirrors #1 and #5

The plasma was produced by an electron cyclotron resonance (ECR) discharge in deuterium in a double-mirror magnetic configuration with maximal magnetic field strength near 0.2 T in magnetic mirrors and magnetron frequency 2.375 GHz [5]. It was shown earlier [1] that such plasma does effectively remove carbon films deposited on metallic surfaces.

The samples were fixed on the water-cooled holder centered along the device axis and brought into the discharge chamber through a vacuum shutter. The magnetic field lines cross samples along the surface normal. The electron density and temperature of plasma in the region of the holder position was near 6×10^{15} m^{-3} and 3-5 eV, correspondingly, according to measurements by electrostatic probes. Before and after cleaning of the contaminating film both samples were weighed within accuracy 20 μg. The cleaning procedure was carried out step by step, with regular *ex situ* control of the spectral reflectance in the wavelength range 220-650 nm. For these particular samples two regimes of film cleaning were applied: (a) without biasing the holder (like in [1]), i.e., when during first 130 min for sample #1 and 140 min for sample #5 the ion energy was defined by the sheath potential only, e.g., not exceeded ~15 eV and thus the chemical erosion was the main mechanism of removing the carbon-based film; (b) with biasing holder to –300 V, e.g., when starting from 130 min for #1 sample and from 140 min for #5 sample the deuterium ion energy much exceeded the threshold of the physical sputtering of carbon and any other contaminant material.

The time dependences of reflectance recovering at two wavelengths for both samples are shown in Fig.6. These data demonstrate that the characteristics of films that appeared on these samples are very different. Namely, the film on the #5 sample was thicker (because the interference effects are seen) but softer than the film on the #1 sample. This follows from the fact that the rate of reflectance recovering for the #5 sample during the cleaning regime (a) was much faster in comparison to the #1 sample. However for both samples the recovery was stopped after about one hour cleaning time. For the #5 sample this "saturation" of mirror recovering (in the time interval 60-140 min) is probably due to practically full disappearance of the carbon film deposited inside the LHD vessel, and the remained film was the one connected with washing the sample in an ultrasonic bath after the finish of polishing, before installation inside LHD. This lowest contaminating layer was gradually disappearing, starting from the time 140 min, i.e., after ion energy was increased by ~300 eV. For the #1 sample the intermediate saturation level behaved in the way like the rest film consisted of not one but two layers: the upper which disappeared by ion bombardment during 130-145 min was probably the rest of the layer deposited in LHD, and the lower one which had the thickness and composition close to the layer that was initially on all samples (due to washing in an ultrasound bath) and maintained on the #5 sample after 1st stage of conditioning. The practically full recovering of the spectral reflectance was achieved for both samples after about 60 min bombardment with 300 eV energy ions.

4. Discussion and conclusion

The data obtained at this stage of experiment demonstrate that the location of the mirror samples inside the LHD vacuum chamber is very important factor that determines the rate of mirror degradation. The mirror #3 located close to the plasma confinement volume and quite distant from the divertor regions was cleaned from the contaminating film that appeared as a result of rinsing samples in the ultrasonic acetone bath. Besides, this mirror was even slightly etched by plasma particles at the depth ~0.15μm. After exposure in LHD the reflectance of this mirror samples significantly increased in comparison with the initial reflectance value and according to profilometry and SEM data, this sample saved the very smooth surface. This is in agreement with data of modeling experiments with mirrors of similar material. Namely, as was shown in [1], the reflectance of stainless steel mirror begin to degrade after depth of sputter erosion exceeds ~0.3μm.

Mirror #1, fixed close to the divertor region with graphite tiles as the divertor plates, becames coated by the film of complicated composition. The thickness of film deposited on the collector #1 measured by AES [2,3] and on the mirror sample #1 by RBS and ellipsometry are in a quite good agreement each other. Namely, the thickness ~40 nm can be estimated from RBS, ~30 nm from AES data (Fig. 3a in [3]) and ~26 nm according to ellipsometry. Similarly, rather good agreement was found for deposited film thickness measured on samples #5: by AES ~50 nm, (Fig. 3e in [3]), by RBS ~100 nm, and estimated from ellipsometry and spectroscopy measurements ~50 nm.

As was mentioned above, the carbon film thickness on the mirror sample #5, fixed deeply in the port, was much thicker according to optical measurement (Fig.2) and results of cleaning (Fig.6), however it could not be measured by ellipsometry just after taking out of LHD vessel because of very low reflectance at 632.8 nm. In the process of film cleaning (Fig.6) the ellipsometry of the sample #5 was applied to obtain the optical characteristics and film thickness at some intermediate phase of the

cleaning procedure. Then these data were used to calculate the spectral reflectance at different stages of cleaning and to compare the calculated values with spectral reflectance measured in the course of cleaning. In Fig.7 the results of calculations are shown by lines and measured reflectance – by symbols. As a parameter of symbol sets the time duration of the cleaning procedure (Fig.6) was taken, and parameter of lines – the thickness of carbon film when providing the calculations (these film thicknesses are indicated in Fig. 7 near every curve). When providing these calculations the wavelength dependences of carbon film n_f and k_f values were taken the same as for calculations of data published in [6] for the carbon film deposited on molybdenum mirror in the arc discharge between two graphite electrodes. The best fitting of calculated data required some variations of supposed optical indices of film (n_f and k_f), in the limit ±10% relatively to the base values, when calculating the spectral reflectance for every stage of cleaning indicated in Fig.7. The need to modify the n_f and k_f values when fitting the different film thickness on the #5 mirror sample is an evidence of a definite depth inhomogeneity of the deposit appeared on the mirror sample #5 during exposure in LHD.

The values of n_f and k_f indices found for deposit on sample #1 within the framework of the simple model (i.e., n=2.5, k=0.33) are also in a quite good correspondence with values characteristic for a carbon film that was evaporated by an arc discharge between two graphite electrodes (n=2.6, k=0.35 [7]) but very differ from indices measured for the film grown on the window of the JT-60U tokamak (n=1.8-2.0, k=0.17-0.15 [8]).

The resistance of deposited films to impact by low energy D ions is quite different as data shown in Fig.6. The cleaning process demonstrates that the film on the #1 sample was significantly harder than that on the sample #5. This is probably the result of high percentage of iron in the composition of the deposit. The SEM photos demonstrated that the film on the #5 sample was strongly inhomogeneous in comparison to the quite homogeneous film surface on the #1 sample.

It is interesting to note that the film appeared on all three sample mirrors due to rinsing in an ultrasonic bath was not too strongly modified on sample #5 during exposing it in LHD and was still a cognate object near the finish of the cleaning procedure. In Fig.8 we compare the spectral reflectance for samples #1 and #5 at the time of cleaning t=145 min with reflectance for all samples before installation into LHD (the same as curve "initial" in Fig.2). The symbols are the measured reflectance and lines – the calculated values with assumption that optical indices of the contaminating film are the same as for the carbon film deposited in the arc discharge between graphite electrodes [7]. As seen, the thickness and optical properties of the film that remained on the sample #5 after this intermediate time of conditioning (t=145 min) are in an excellent agreement with the film which was initially on all samples due to rinsing them in an ultrasonic bath. Thus, we can state that before installation into the LHD vessel all samples had a film of ~11 nm thick.

At the same time there is no similar agreement for the sample #1. The film remained here after 145 min cleaning procedure was ~50% thicker (~15 nm) and had different optical constants, what follows from the difference between measured points (rhombuses) and the curve calculated for the same n_f and k_f values as two other curves at this graph.

Thus, we can conclude that during the exposition in LHD the sample #3 was practically fully cleaned from the contaminating layer, and on the samples #1 and #5 this layer was over-deposited by another layer. This new layer practically did not

change the characteristics of the initial film on the sample #5 ("soft" deposition in LHD), as it is seen from Fig.8: the properties of the rest layer on this sample at this stage of cleaning are very much the same as those which all samples had before installation into LHD (the curve "initial" in Fig. 2 and curves for samples #1 and #5 in Fig. 8). At the same time, the residual film on the sample #1 has significantly different optical indices in comparison with indices of the initial layer that existed on all samples before installation into LHD. It follows from the latter that the process of deposition of contaminating film on mirror #1 was accompanied by modification of the film that existed on the mirror surface initially, before installation of mirrors into LHD ("hard" deposition in LHD).

After full finishing the cleaning procedure the spectral reflectance of both mirrors (#1 and #5) became very close to what is shown in Fig.2 by squares as an example of typical SS mirror which was polished, rinsed in an ultrasound bath, and cleaned by low energy deuterium ions.

The mechanism which provided the cleaning of the sample #3 from the initial contaminating film and the maintenance of a high surface quality with corresponding high reflectance was not identified yet.

Basing on the above described results we can make the following conclusion:

The correct choice of diagnostics mirror location inside the LHD vacuum chamber with graphite tiles protecting the vessel wall in the divertor area is a quite responsible procedure. It is evident that the mirror fixed at the same position as the sample #3 will maintain its optical properties for a long period of LHD operation. Such mirrors can be used for observation of those parts of plasma or inner surfaces (e.g., divertor plasma, divertor plates) that are not seen directly through the diagnostic ports.

In the case of inappropriate choice of the mirror position the cleaning of contaminating carbon-based deposit on the mirror surface can become a quite difficult problem because the biasing of the mirror holder to several hundred volts would be required.

Using the fitting method for measured spectral reflectance behavior during different stages of the cleaning procedure, the thickness and optical indices of the carbon-based contaminating film appeared on the mirror surface can be estimated.

The behavior of mirrors in conditions when the boronization procedure is used would be very desirable.

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Figure captions

Fig.1. The scheme of locations of SS mirror samples inside the LHD vessel.

Fig. 2. Spectral reflectance of SS mirror samples before (marked as "initial") and after exposure inside the LHD vessel (curves marked as #1, #3, and #5). Squares show the typical spectral reflectance of a SS mirror subjected to cleaning by low temperature deuterium plasma after polishing and washing in an ultrasonic acetone bath.

Fig. 3. The photos of samples after they were taken out of the LHD vacuum chamber.

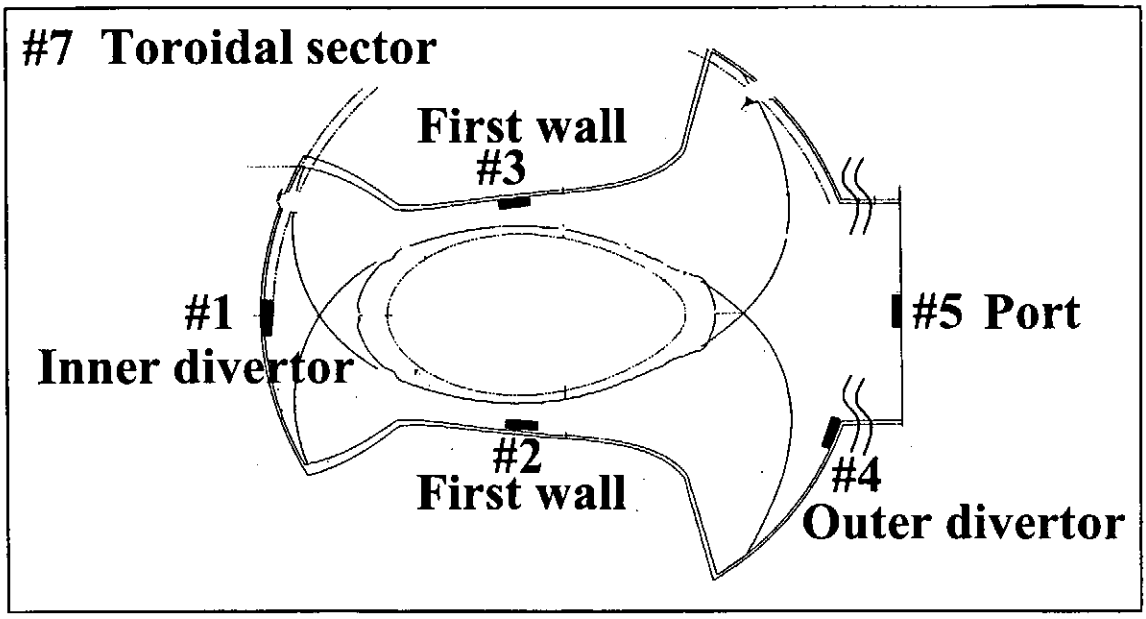
Fig. 4. Results of profilometry measurement of the surface morphology of the sample #3 after exposing in the LHD.

Fig. 5. RBS spectra for collectors and mirror samples.

Fig. 6. The recovery of reflectance (normal incidence, wavelengths 220 nm and 650 nm) of samples #1 and #5 due to deuterium ion bombardment. Up to t=130 min for sample #1 and up to t=140 min for sample #5 the sample holder was grounded, i.e., the energy of ions was <15eV. After that times the samples were exposed to ions accelerated to ~300 eV by negatively biased holder.

Fig. 7. The comparison for the sample #5 of the measured reflectance values (symbols) and calculated reflectance values (curves), correspondingly, for different time moments of cleaning procedure with the film thickness d_f as a parameter.

Fig. 8. The measured (symbols) and calculated (curves) spectral reflectance values: for samples #1 and #5 after t=145 min cleaning procedure; for sample #3 – before installation of samples into LHD (curve "initial" in Fig. 2). After fitting the initial reflectance data for sample #3 (solid line) we took the same $n_s(\lambda)$, $k_s(\lambda)$ for the stainless steel and $n_f(\lambda)$, $k_f(\lambda)$ for the carbon film on samples #1 (dotted line) and #5 (dashed line).



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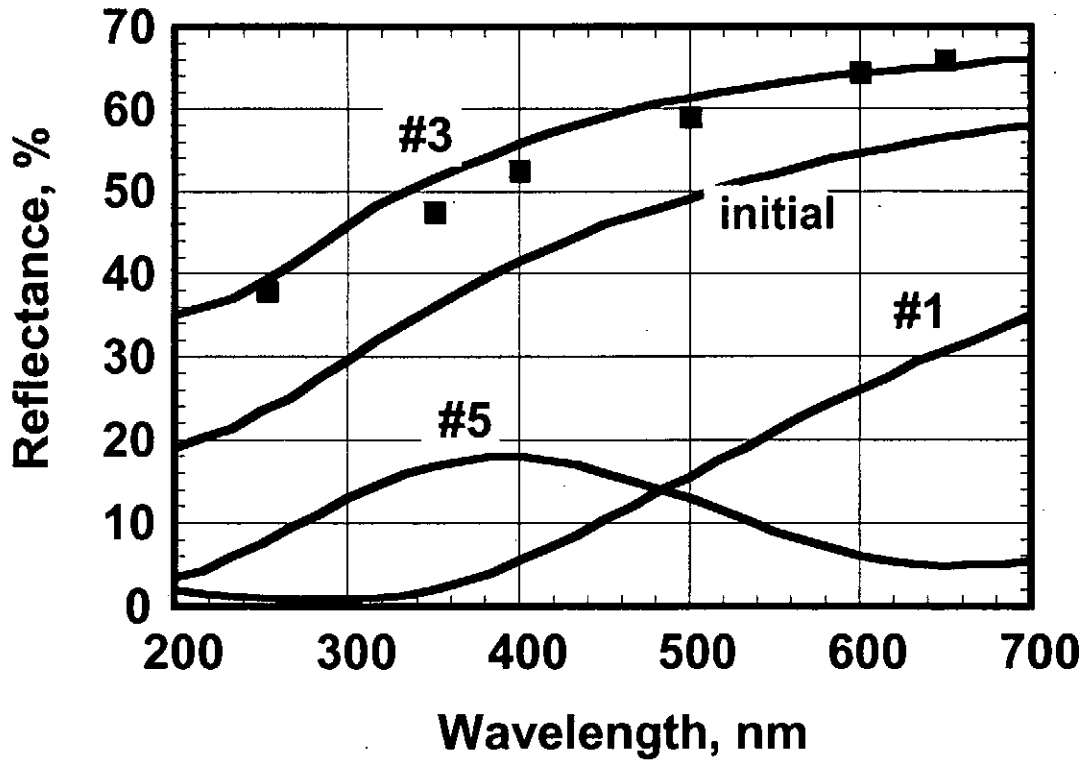

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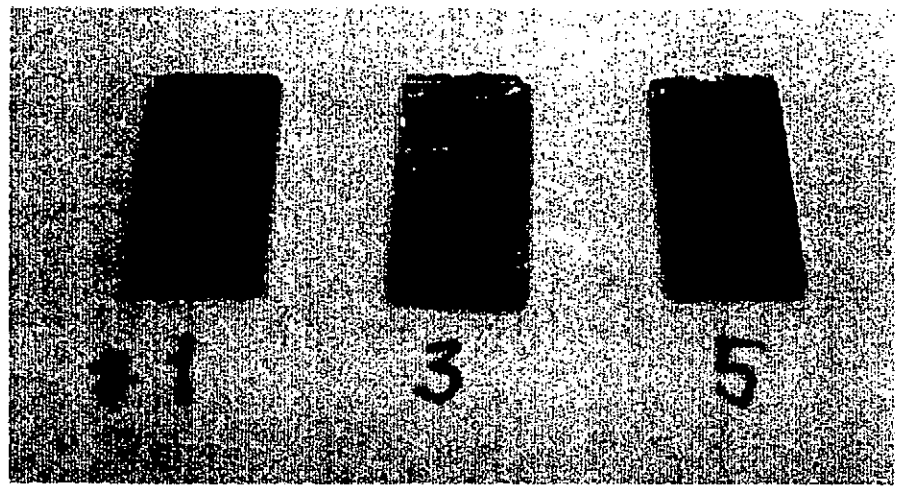

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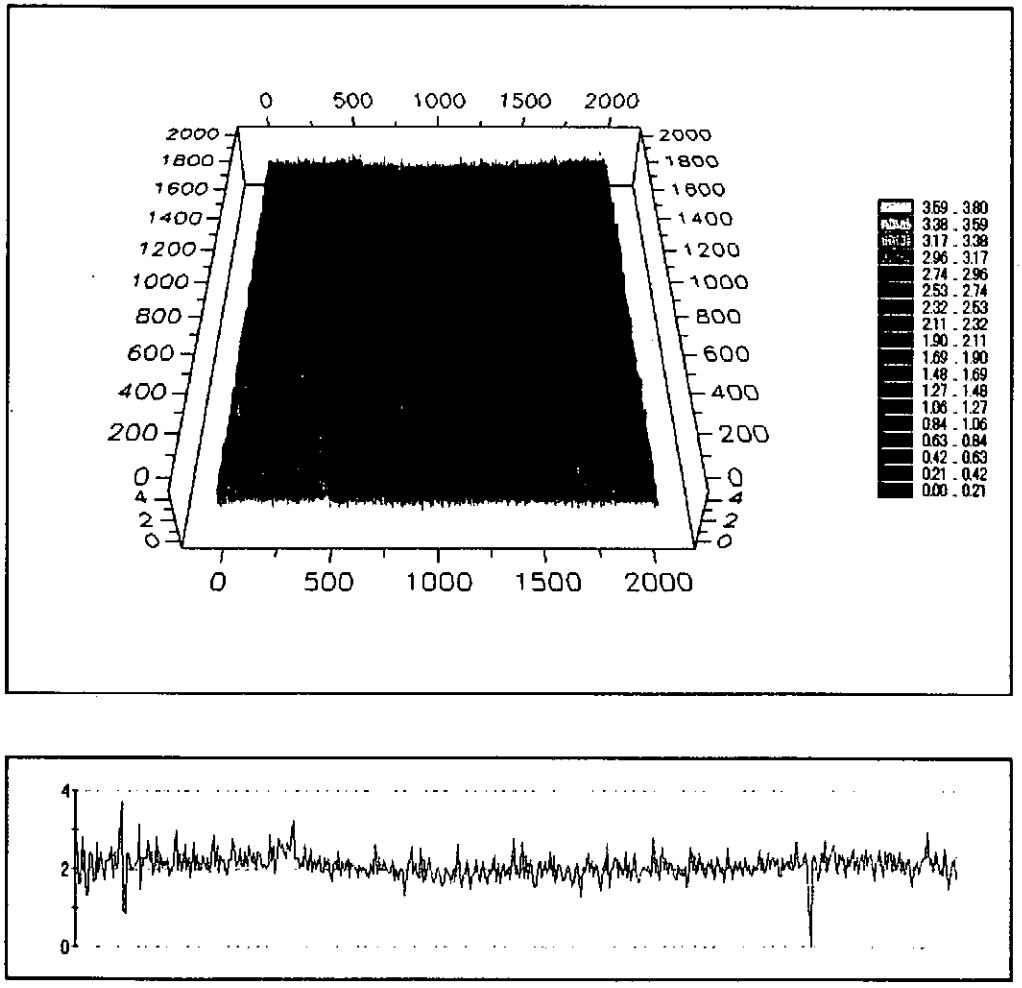

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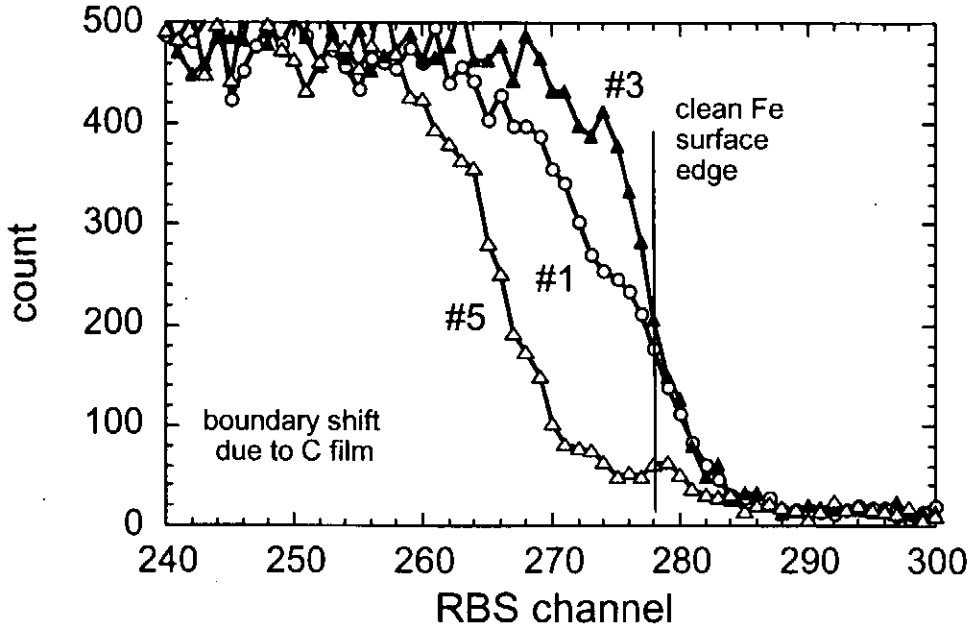


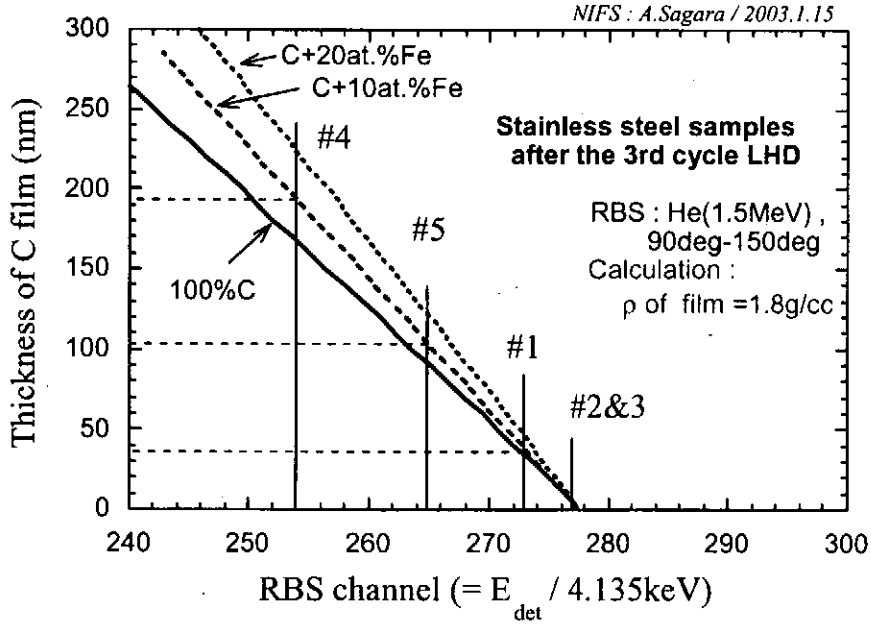
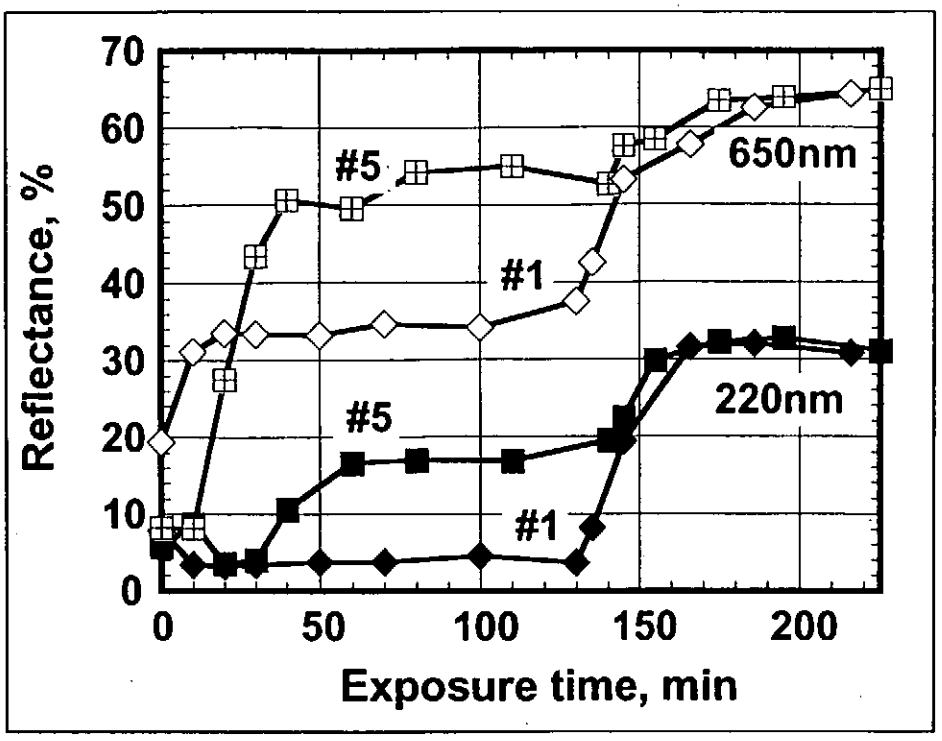


Fig. 5. RBS spectra for collectors and mirror samples,
and the C film thickness calculated as a function of RBS channel.



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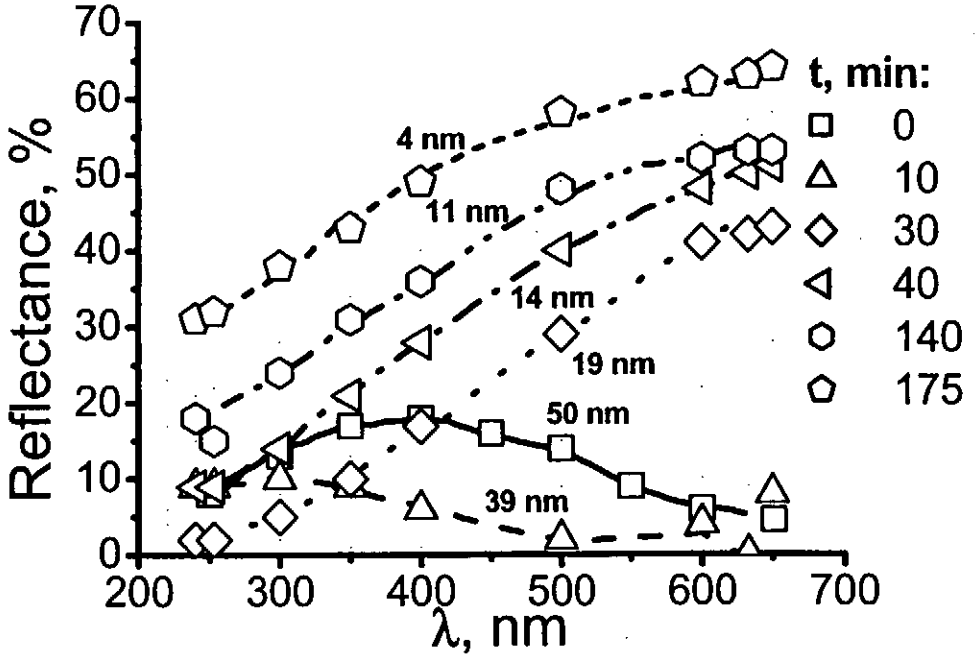

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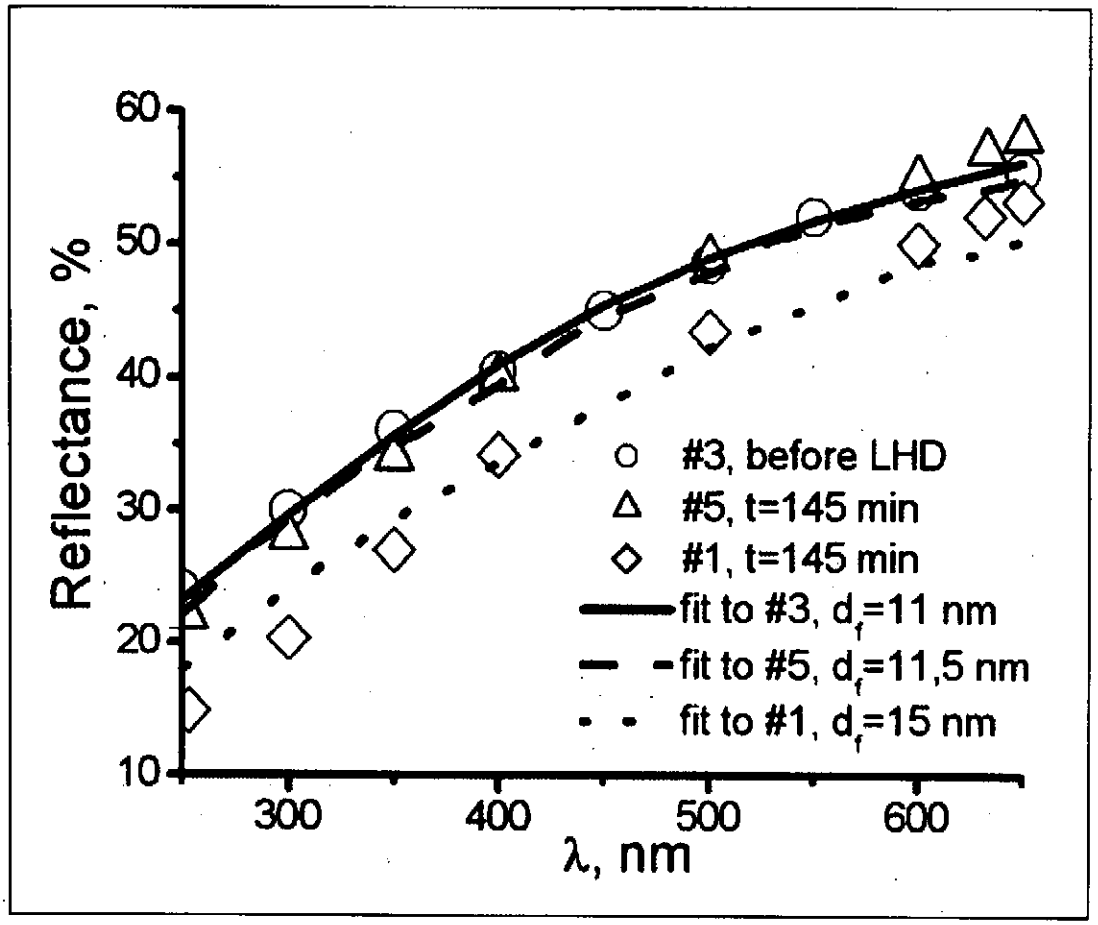

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