

Structure, Phase Analysis and Component Composition of Multilayer Films Depositing in Tokamak T-10

M.I.Guseva, V.M. Gureev, L.N. Khimchenko, B.N. Kolbasov, K.Yu. Vukolov

Kurchatov Institute, Moscow, Russia

e-mail contact of main author: kolbasov@nfi.kiae.ru

Abstract. The films formed on the internal surface of the T-10 tokamak vacuum chamber and on the stainless steel mirror-specimens located inside the T-10 tokamak upper port during 2002 and 2003 experimental campaigns have been studied using X-ray diffraction analysis, Rutherford backscattering, scanning electron microscopy and elastic recoil detection method. Before the 2003 experimental campaign the graphite ring diaphragm was removed from the tokamak and a new movable limiter made of RGT-91 graphite was installed. Mirror surfaces could be screened during chamber conditioning and exposed to plasma only during working discharges. The films formed on the chamber walls in both campaigns were “soft”, reddish-brown and had a multilayer structure. Few hundreds of thin (<100 nm) layers were detected in cross sections of such films. X-ray diffraction analysis suggests that these films have amorphous structure and contain up to 10 % C-60 (fullerens) with lattice constant of 12,086-14,055. D/C atomic ratio in the films increased from 0.66 in 2002 to 1.7 in 2003. The “soft” film formation was caused by the plasma-wall interaction during the vacuum chamber conditioning. The films on mirrors were thinner than those on the vacuum chamber walls and, as a rule, semitransparent. They consisted of 10-12 layers formed during a campaign. Layer thickness depended on the total film thickness (on the exposition site). The surface structure of these films was homogeneous and smooth, without any signs of physical sputtering. The films deposited on the mirror surface exposed to plasma only during working discharges in 2002 were “hard” with $D/C = 0.26$ and those formed in 2003 were “soft” ($D/C = 1.55$). Two crystalline phases with interplanar spacings of 0.359 and 0.304 nm at the Wulf-Bragg diffraction angles 2θ of 24.8 and 28.8° respectively were revealed in a diffractogram of a hard film. Deposition of a “soft” film on a mirror open to exposure only during working discharges is caused by the increase of the plasma-wall interaction in 2003.

1. Introduction

Two types of re-deposited hydrocarbon films (homogeneous and globular) differing in their surface structure and internal texture were found inside the T-10 tokamak vacuum chamber [1]. Other films similar in their microstructure to the homogeneous ones have formed during the same experimental campaigns on the smooth plasma facing surfaces of the stainless steel mirror-specimens located inside the upper stub pipe of the T-10 tokamak vacuum chamber [2]. At the same time, the films formed on the chamber walls and mirrors essentially differ in hydrogen isotope content.

Basic characteristics of the films deposited on the vacuum chamber walls and mirrors during the 2002 and 2003 experimental campaigns of T-10 tokamak are compared in this study.

2. Experimental Technique

The 4 mm thick 04Kh16N11M3T (SS316) stainless steel mirrors that measured 9 mm by 9 mm were used in the experiments. They were exposed in the upper diagnostic port of the limiter cross-section of the T-10 tokamak, 22 cm distant from plasma. Some mirror surfaces were shielded during chamber conditioning by a shutter and were exposed to plasma only during working discharges. During the 2002 and 2003 experimental campaigns the mirrors were located in the same positions facing plasma. The films which were formed on the vacuum chamber walls and flaked out from it were collected from the vacuum chamber bottom.

The typical parameters of T-10 discharge are following: working gas is deuterium, plasma current is in the range 200–400 kA during 1 s, toroidal field is 2.8 T, electron temperature in the plasma column center $T_e(0) = 1$ keV during ohmic heating stage. It is increased up to 2.5 keV at the stage of electron cyclotron resonance heating. The electron density averaged along the central chord $n_e = 1\text{--}6 \times 10^{19} \text{ m}^{-3}$. The ion temperature in the plasma column center is 450–600 eV at both stages. Usual configuration with the ring diaphragm and the movable limiter made of graphite MPG–8 was used in the T–10 tokamak in 2002. In 2003 the ring diaphragm was moved away, and only movable limiter made of graphite RGT –91 was used. The change of central plasma parameters resulted from this modification was small, but the parameters at the plasma column periphery changed significantly and the intensity of plasma–wall interaction greatly increased.

Every campaign started from the vacuum chamber baking up to 200 °C, chamber surface cleaning by inductive H₂ or D₂ discharges, and He or Ar glow discharges. During experimental campaigns, vacuum chamber conditioning was used practically every night. The data on duration of the vacuum chamber conditioning modes and working plasma discharges are presented in Table 1.

TABLE I: BASIC DATA ON VACUUM CHAMBER CONDITIONING MODES AND WORKING DISCHARGES.

Year	Baking, h	Inductive discharges, h	Glow discharges, h	Working discharges, s
2002	897	35 (H ₂), 270 (D ₂)	86 (He)	1620 (H ₂)
2003	367	24 (H ₂), 79.5 (D ₂)	19 (He), 2 (Ar)	212 (H ₂)

The film thickness was measured using TENCOR Instruments profilometer and Rutherford backscattering (RBS) analysis. The RBS in combination with the resonance elastic scattering was measured in the Van de Graaf accelerator in which a 2-MeV proton beam was backscattered from a specimen under study at 170° to its surface. The distribution of hydrogen isotopes in depth of films was measured using the elastic recoil detection analysis, in which a 1.9-MeV He⁺-ion beam hit a specimen. Phase film composition was determined studying X-ray diffraction in the Dron-4 diffractometer.

3. Experimental Results and Their Short Discussion

The deposited films were observed on the mirrors and vacuum chamber walls after T-10 tokamak operation. It means that redeposition predominated over erosion on these surfaces under the tokamak operation conditions both in 2002 and 2003. The surface of looked shining and smooth. The film colour varied from golden-crimson to violet-green. The films deposited on the vacuum chamber walls are yellow and reddish-brown. The screened parts of the mirror surfaces in visual perception practically did not change. The films have very homogeneous fine-dispersed structure with inclusions of isolated 1–3-μm spherical particles and flakes up to 25 μm in size. They originated from brittle destruction of the limiter under plasma disruptions. There are tracks of hard microparticles and molten metal microdroplets on mirrors. These films are classified as polymer-like re-deposited hydrocarbon a –C:H films [3].

Microstructure of the end of a 140 μm thick multilayer film deposited on the vacuum chamber wall in the 2003 campaign is presented in Fig. 1. According to RBS data, the deposits on the open parts of the mirrors basically consist of carbon and hydrogen isotopes with 8-16 at.% oxygen.



FIG.1. Microstructure of an end of a film deposited on the vacuum chamber wall in the 2003 campaign

A diffractogram fragment for a reddish-brown film is shown in Fig. 2. The diffuse image of the spectra in the range of the Wulf-Bragg diffraction angles $2\theta = 25^\circ$ and $10-15^\circ$ suggests that the film had amorphous structure. According to a calculation of the quantitative phase composition which was performed using the PHAN% code, attached to the Dron-4 diffractometer, the reddish-brown films contain up to 10 vol.% C-60 (fullerens) with the lattice spacing in the range from 12.86 to 14.055 and 90 vol.% graphite with the lattice spacing in the range of 8.688–9.020. A similar conclusion on the existence of fullerenes in the films formed in T-10 tokamak was drawn in the paper [3] on the basis of photoluminescence excitation spectra studies for these films. Compounds of C, H, N and O ($C_9H_8N_2O_2$, $C_{14}H_{10}O_2$, $C_4H_5N_3$ and $C_{16}H_{16}N_2O_4$) were observed at the angle $2\theta = 28.8^\circ$.

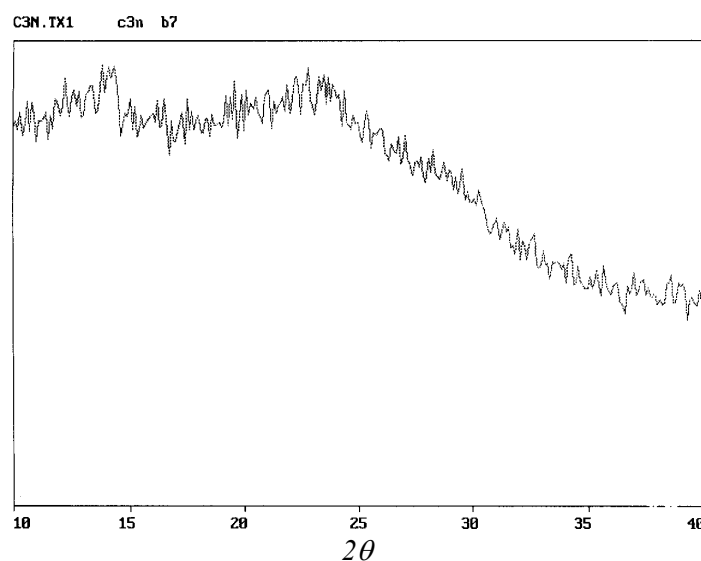


FIG. 2. Diffractogram fragment for a reddish-brown film formed on vacuum chamber wall.

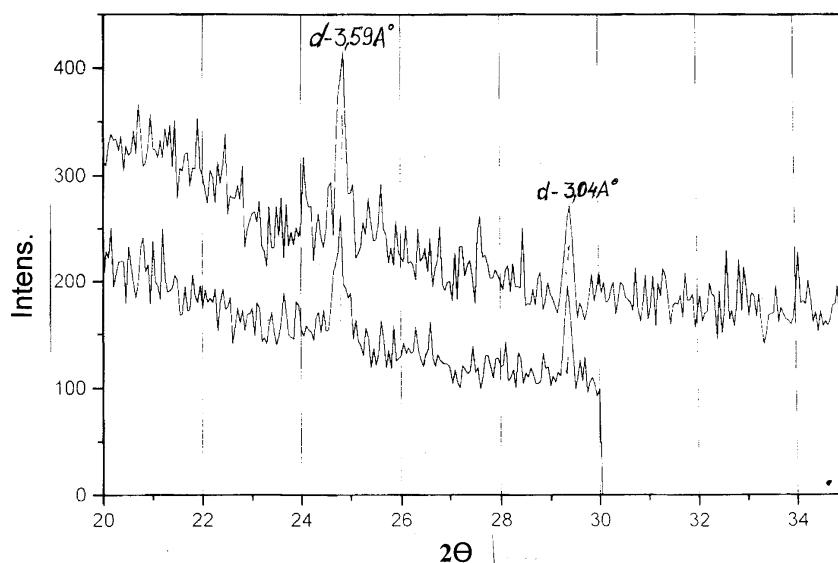


FIG.3. Diffractogram fragment for a film deposited on a mirror specimen in 2002.

A diffractogram fragment for a film deposited on a mirror specimen in 2002 is presented in Fig. 3. Its analysis shows that all the carbon is chemically bound with deuterium. Two crystalline hydrocarbon phases with interplanar spacings of 0.359 and 0.304 nm at the Wulf-Bragg diffraction angles 2θ of 24.8 and 28.8° respectively were revealed in the diffractogram. Hence, formation of the hydrocarbon structure containing crystalline phase, was observed on a mirror, screened with a shutter at vacuum chamber conditioning during 2002 experimental campaign.

Deuterium and protium atom distributions in depth in films deposited on mirrors in 2002 (a) and 2003 (b) are shown in Fig. 4. Deuterium in these films is distributed sufficiently uniformly, except near-surface zone where the concentration decrease takes place. Protium is available only in near-surface layers of the films where deuterium concentration reduction is observed. Deuterium concentration in the film deposited in 2003 is almost 3 times as many as in the film deposited in 2002.

Deuterium and protium concentration profiles in depth of reddish-brown films formed on vacuum chamber walls in experiments of 2002 (a) and 2003 (b) is presented in Fig. 5. The analysis of Figures 4 and 5 is given in Table II. Protium in the reddish-brown films formed on the chamber walls, unlike films on mirrors, is distributed along the whole film thickness.

TABLE II: BASIC PARAMETERS OF FILMS DEPOSITED IN T-10 TOKAMAK DURING EXPERIMENTAL CAMPAIGNS OF 2002 AND 2003.

Film substrate	Year	Film thickness, μm	Integral concentration, 10^{20} m^{-2}		RBS, at. %		D/C	Comment
			D	H	C	O		
Mirror	2002	0.3	65	5.8	69	8-13	0.26	Mirror is screened during conditioning
Mirror	2003	0.7	180	9.3	34	8-13	1.55	
Wall	2002	5	120	23	49	16	0.66	Reddish brown film
Wall	2003	140	192	69	34	8-16	1.70	

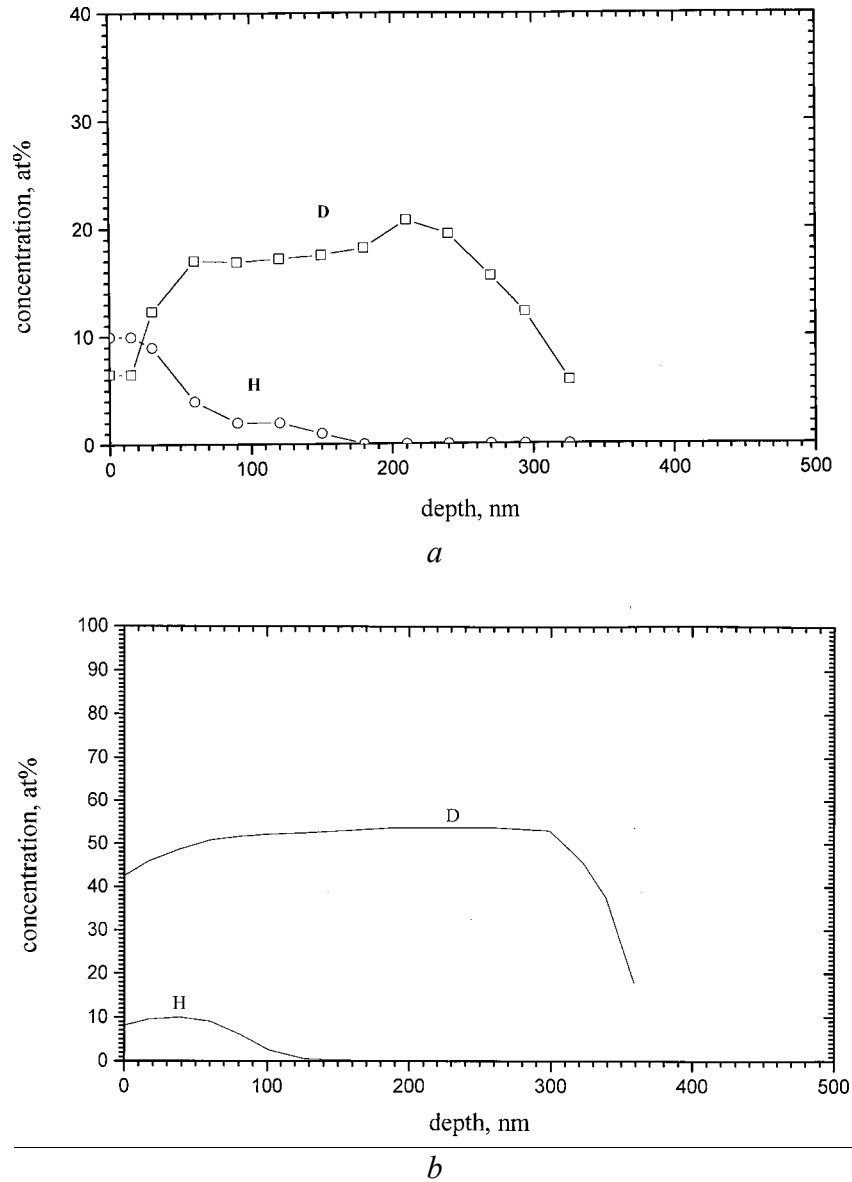


FIG. 4. Deuterium and protium atom distributions in depth of films deposited on mirrors in 2002 (a) and 2003 (b)

The following conclusions can be drawn from the consideration of Table II:

1. The film, deposited on a mirror-specimen surface, closed by a shutter during vacuum chamber conditioning in 2002 experimental campaign, was “hard” ($D/C = 0.26$). The film formed in the same experimental campaign on the chamber wall was “soft” ($D/C = 0.66$). Hence, the “soft” film formation is caused by the plasma-surface interaction during vacuum chamber conditioning.
2. The film deposited in similar 2003 experiments on the mirror-specimen, closed by a shutter during vacuum chamber conditioning, was “soft” ($D/C = 1.55$). This film has characteristics similar to those of a reddish-brown films formed on the vacuum chamber walls during the same campaign ($D/C = 1.7$). Such an effect apparently is caused by increased intensity of plasma-surface interaction. The plasma column parameters in 2003 campaign did not change appreciably in comparison with the 2002 campaign, but parameters of periphery plasma could change essentially.

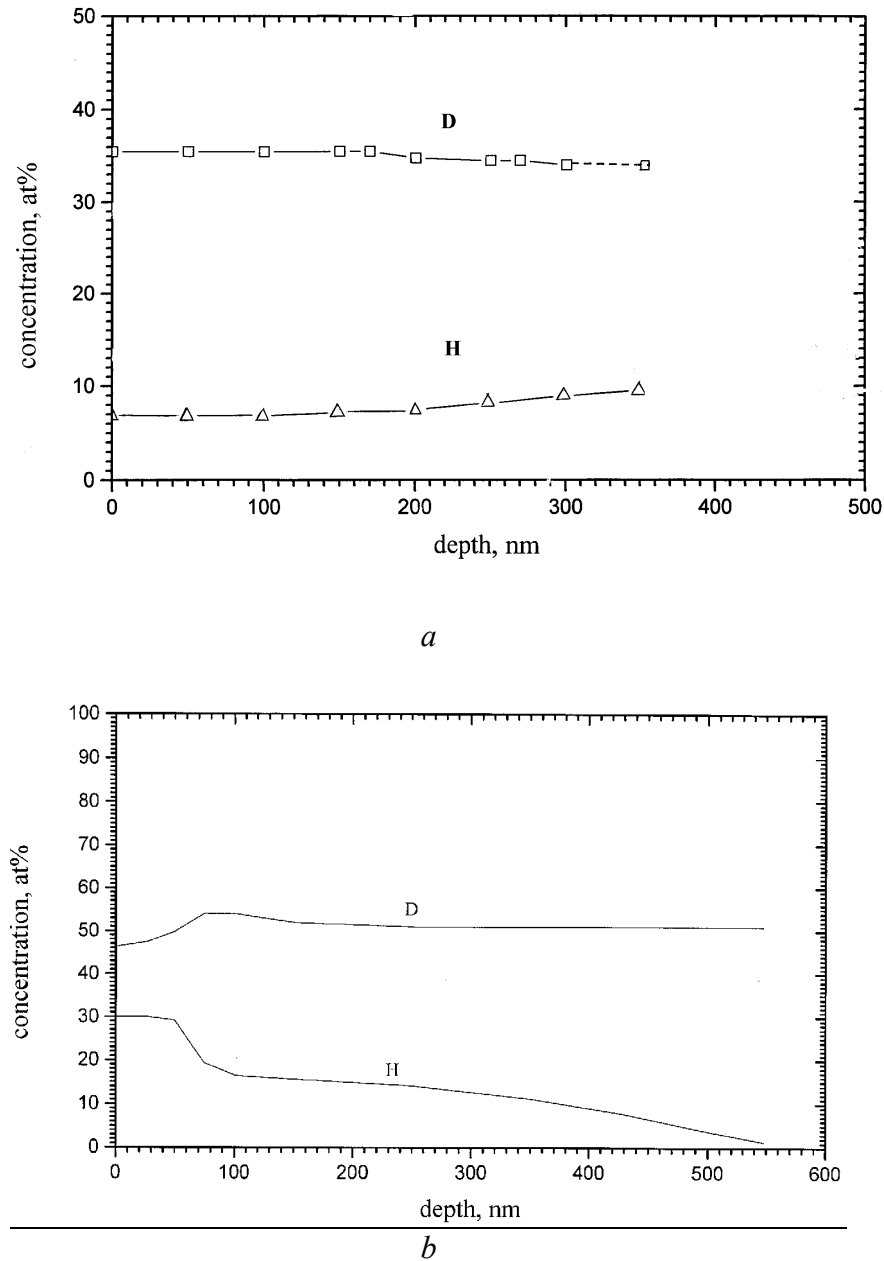


FIG. 5. Deuterium and protium concentration profiles in depth of reddish-brown films formed on vacuum chamber walls in 2002 (a) and 2003 (b) experimental campaigns.

The deposition rate onto the mirror, closed by a shutter during the vacuum chamber conditioning, was calculated for the on the basis of known exposure duration and film thickness. It was 0.2 nm/s in 2002 and 4 nm/s in 2003. Simulation studies performed in an accelerator, where hydrocarbon films, which had microstructure and internal texture similar to those of the films formed in tokamaks, were synthesized, have shown that “soft” films are not formed at irradiation temperature of 400 °C [4].

This study has shown that, in principle, formation of “soft” films with high hydrogen isotope content in tokamaks equipped with carbon plasma facing elements can be suppressed by selection of vacuum chamber wall temperature during the wall conditioning.

4. Conclusions

1. The process of film deposition in T-10 tokamak dominates over their erosion.
2. Screening of mirrors at tokamak conditioning under normal experimental conditions (during 2002 operation campaign) resulted in deposition of “hard” deuterocarbon films on the mirror surface.
3. “Soft” films form on the T-10 tokamak vacuum chamber walls during the chamber conditioning.
4. During 2003 experimental campaign, when the ring diaphragm was removed and a new limiter made of RGT-91 graphite was installed, “soft” films with $D/C > 1$ deposited on the vacuum chamber walls and mirror surfaces, screened at the chamber conditioning by a shutter.
5. Hydrogen isotope accumulation in the films is determined by the processes of plasma-surface interaction and by parameters of the plasma column on its periphery.
6. Formation of “soft” films may, in principle, be avoided by keeping vacuum chamber walls at temperature about 400 °C [4].

The authors would like to thank P.V. Romanov for useful discussions and interest to the study.

References

- [1] GUSEVA, M.I., et al., “Microstructure features and deuterium content in carbon globular films re-deposited in T-10 tokamak”, Problems of Atomic Science and Engineering, Ser. ‘Thermonuclear Fusion’, Kurchatov Institute, Moscow (2001) No. 1, pp. 3–12 (in Russian).
- [2] VUKOLOV, K.Yu., et al., Controlled Fusion and Plasma Physics (Proc. 30th EPS Conf. St. Petersburg, 2003), ESA, Vol. 27A, p. 2,76.
- [3] SVECHNIKOV, N.Yu., et al., “Spectroscopic Studies of Homogeneous Thin Carbon Erosion Films on Mirrors and Flakes with a High Deuterium Content Formed in Tokamak T-10” Fusion Technology (23 Symp. Venice, 2004).
- [4] GUSEVA, M.I., et al., “Study of co-deposited carbon layers and of mixed (W+C) layers on tungsten and graphite in a plasma accelerator”, Vacuum **67** (2002) 253.