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Spectroscopic studies of homogeneous thin carbon erosion films on mirrors and flakes with a high deuterium content formed in tokamak T-10

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Abstract

Redeposited hydrocarbon films on plasma facing elements in tokamaks accumulate hydrogen isotopes. In the present study such films were made to redeposit on stainless steel mirror substrates as thin films and without any substrate as bare flakes with high deuterium content, under deuterium-plasma discharges inside T-10 tokamak vacuum chamber. These films were subjected to spectral characterizations through Fourier-transform infrared (FT-IR), electron paramagnetic resonance (EPR), and photoluminescence techniques. IR spectra showed the presence of two main deuterium states as observed by the $CD_{2,3}$ sp^3 stretching modes at 2100–2200 cm^{-1} and the CD_2 sp^3 bending modes at 600–1100 cm^{-1}. Among these, CD_3 stretching mode at 2217 cm^{-1} may serve as a control for deuterium desorption during the cleanup process of the reactor. As a comparative measure, C60 films were also studied, the luminescence excitation spectrum of which showed similarity in peak positions with tokamak bare flakes pertained to sp^2 luminescence centers. The observed spectral differences are mainly due to more localized sp^2 states for C60 and sp^3 states for tokamak flakes. EPR spectra of the bare flakes showed the defective states with a high spin density, $\sim 10^{19}$ cm^{-3} which serve as luminescence quenching centers, and provide a path for hydrogen isotopes adsorption.

Keywords: Tokamak; Redeposited hydrocarbon films; Vibrational modes; Photoluminescence; Defects

1. Introduction

The basic requirement for ITER safety is to control and prevent the radioactivity releases not to exceed the environmental release limits. One such control measure is to contain the high volatile tritium within the design

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limit as 330 g [1,2]. During the first 10 years of effective ITER operation about 12,000 deuterium-tritium pulses, equivalent to energy generation of 220 GJ/pulse, should be performed [1,2]. At each pulse ∼0.39 g of tritium is burnt with 2–5 g of tritium might be co-deposited with sputtered carbon atoms and with sputtered beryllium atoms in slight excess amounts [2]. Thus, in a reactor's lifetime an estimated amount between 25 and 65 kg of tritium may be accumulated as erosion products inside the reactor's vacuum chamber prompting its removal after every 60–160 pulses or 20–50 times per year. The search for ways to decrease tritium accumulation rate inside the vacuum chamber is of paramount importance and is a subject of serious and continuous study.

Studies on physical properties of these redeposited hydrocarbon films such as surface states, valence bonds, mechanisms of accumulation, binding energies and desorption temperatures will greatly help to understand the basic nature of hydrogen isotope accumulation in these films. In this work, a series of spectroscopic studies were carried out with the redeposited hydrocarbon reddish-gold films without any substrate (flakes), having a high D/C ratio 0.4–0.5, and H/C ratio 0.1–0.2, thickness 10–30 μm, and with the stainless steel mirrors (SS316) substrates as thin films: SS mirror No.2 (thickness 0.3 μm, D/C = 0.2, H/C = 0.1–0.2), SS mirror No.6 (1.0 μm, D/C = 0.35, H/C = 0.1–0.2), produced in the T-10 tokamak operating in the Kurchatov Institute [3,4]. SS mirror No.2 was exposed only under working deuterium-plasma discharges, but was closed during the training glow and RF discharges with H_2, D_2^+ and He at vacuum chamber wall degassing. The film thickness was measured with a profilometer. The Rutherford backscattering in combination with the resonance elastic scattering was used for analysis of deposits composition. Hydrogen isotopes depth's profiles were measured with elastic recoil detection analysis by using He^+-ion beam with the energy of 1.9 MeV [4].

The obtained results were analyzed and interpreted in terms of valence band electronic structure and defect states, vibrational modes of CD and CH clusters which control the film properties, temperature dependence on deuterium vibrational modes. The discussion is extended to clarify the mechanism of probable accumulation of deuterium through luminescence quenching centers present in these hydrocarbons.

2. Fourier-transform infrared (FT-IR) spectroscopy of thin erosion films on mirrors

The FT-IR spectra were obtained using Bio-Rad IR Microscope (Model: UMA 500) in retro-specular reflection mode with a spectral resolution of 8 cm^{-1} in the mid-IR range 4000–400 cm^{-1} [3]. Fig. 1 (SS mirror No.2) shows the FT-IR spectra for different spots on the multi-colored mirror, from curve 1 (unexposed region covered by the steel screen with a 0.1-mm gap), to curves 2–4 for the exposed regions, only maximum film thickness was measured, i.e. curve 4 corresponds to 0.3 μm. The IR reflectivity is diminishing with thickness as implied by increase in the absorption. The mode near 633 cm^{-1} can be assigned either to a bending δCD_2 sp^3 mode, or to a δCH sp^1 mode (≡C–H acetylene-like mode). Okuyama et al. observed the δCD_2 sp^3 mode at 638 cm^{-1} for d-ethylene on Pd(1 1 0) [5], while Deschenaux et al. found the δCH sp^1 mode at 628 cm^{-1} for acetylene films [6]. Since in the present case the mode at 633 cm^{-1} was found to shift with film growth to 637 cm^{-1} and to saturate, this can be assigned to a δCD_2 mode. Besides, there is a weak bending δCD_2 mode near 1090 cm^{-1}. For stretching νCD_2, νCD_3 sp^3 modes which appear at 2200–2100 cm^{-1}, can be observed only at higher D/C concentrations (discussed further), there is only a weak concave region on the curve 4 in the region of 2100–2200 cm^{-1}. It should be noted that the induced

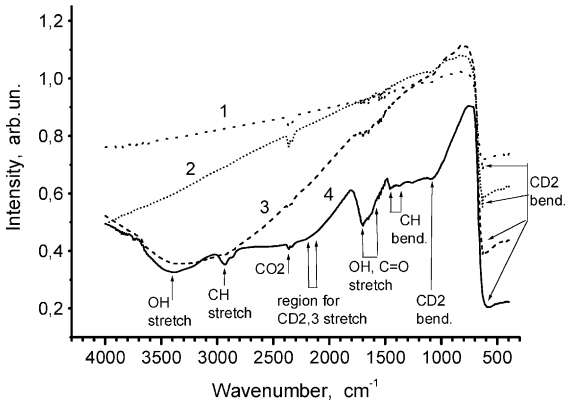

Fig. 1. FT-IR spectra of the redeposited hydrocarbon tokamak film on SS mirror No.2 (0.3 μm, D/C = 0.20, obtained under working D-plasma discharges: curve 1, for unexposed region; curves 2–4, for regions corresponding to the growing film thickness up to 0.3 μm (curve 4).